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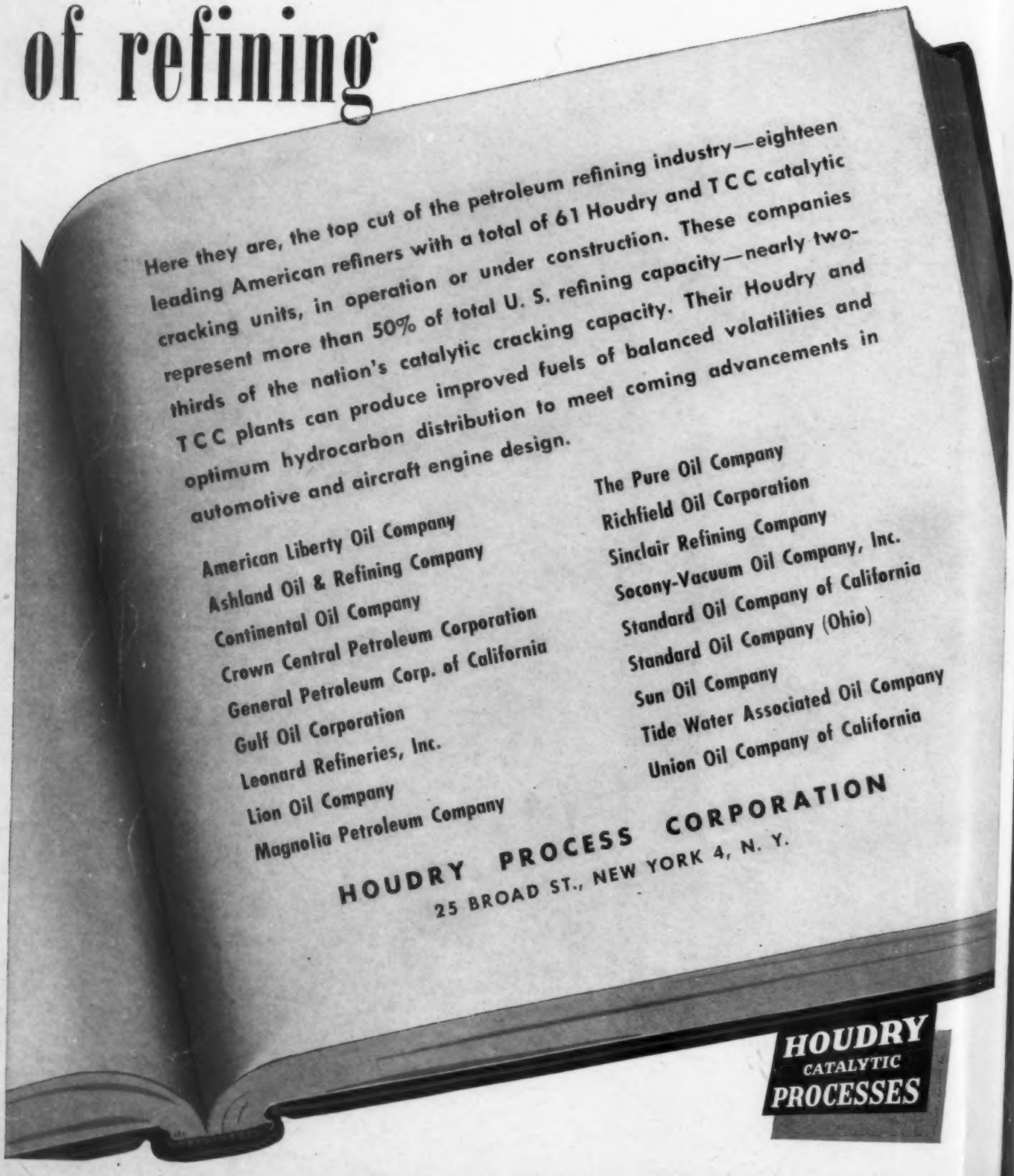

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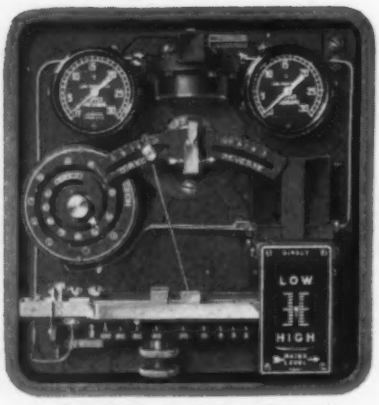

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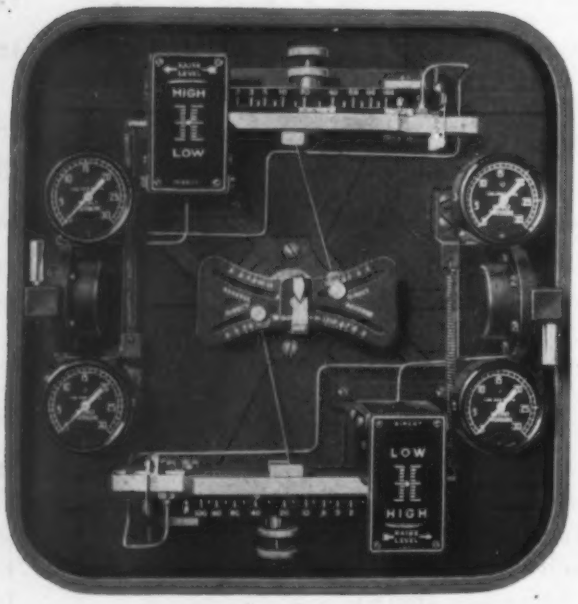

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THE LOOK BOX

THE world over, oil consumption is confounding the economists. Peacetime uses have so outstripped predictions that revised calculations are being applied. These dictate an increased refining capacity, with Europe in line for at least another 500,000 barrels of daily throughput. In the United States the increased capacity is needed especially in the area east of the Rocky Mountains.

Economists' Dilemma

Recent statistics are significant. All-time high point in refinery throughput in the United States was a week in August, 1945, when 5,140,000 barrels daily was charged. But in January, 1947, a winter week and in peacetime, runs stood at 4,917,000 barrels daily, a figure out of line with predictions. In February runs stood for a week at 4,860,000 barrels daily, 5 percent higher than a year previous.

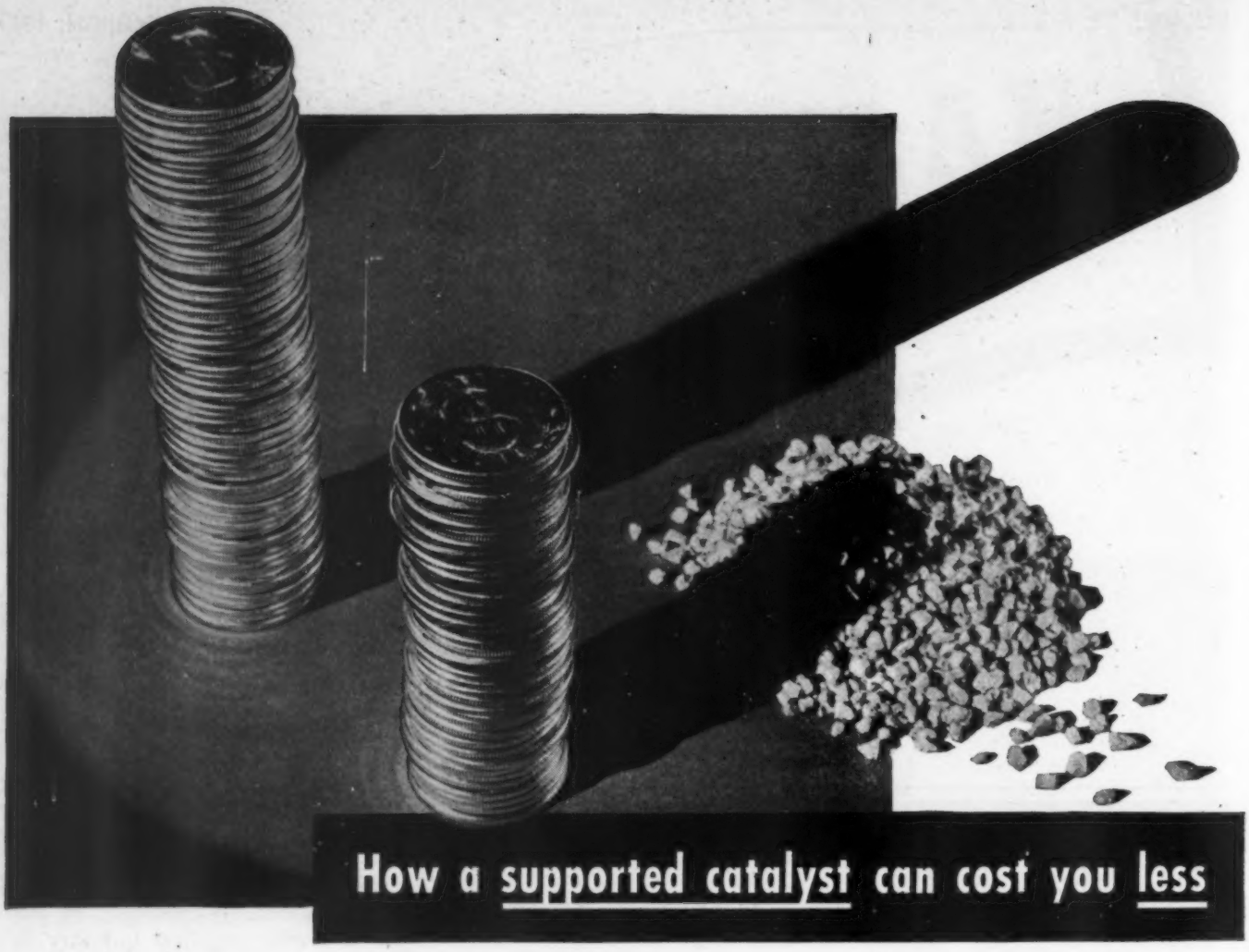
Further analysis shows that runs in California have accounted for so much of the little reduction that exists, that the area east of the Rocky Mountains continues to operate its refining capacity close to full 100-percent throughput. For four weeks ending in mid-January runs in this area were 4,064,000 daily, within 5 percent of what Howard W. Page, economist for Standard Oil Company (New Jersey) considers 100 percent of capacity.

These conclusions as to the refining world picture are based on statistics he used in an interview with R. L. Dudley, publisher of PETROLEUM REFINER in an extensive analysis of all phases of the world oil industry.

Capacity of the domestic refining industry is being expanded, chiefly through modernizations. Only one new plant is slated for the United States. But so many plants have programs for adding new units that the total throughput will expand of necessity, despite the fact that expansion is not the primary purpose of construction. The trend of the last 20 years, fewer plants with higher total throughput, continues.

Europe presents the puzzling situation. Despite its economic plight, the consumption of oil holds so far above calculations that there has been recent shifting of ownership of oil from the Near East fields, so as to permit its consumption in Europe. This consumption will not be possible except through addition of processing units.

The strange phase of the situation in Europe is that its politico-economic programs contribute to the demand for oil. Under a nationalized coal program, English industry has come up short of fuel in a severe winter and the shift to oil is striking. Here is the strange situation in Italy, where coal is imported at $25.00 per ton. Oil with heat value equal to that in a ton of coal can be delivered into Italy for $9.00. A substantial part of the coal production in France is mined by prisoners of war, and their removal will reduce the coal supply, unless immigrants from North Africa are brought in for that purpose.



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Other than the Ruhr, oil is competitive with coal throughout Europe. The disposition of men not to work underground plus other social demands are in part responsible for this, but social programs and personal decisions are as real in economics as are goods on hand.

The prospect in Europe has management of the oil industry in a quandry. Shall it build plants in countries where governments are moving toward collectivism or shall governments of that nature be encouraged to provide this needed capacity? There is no one-word answer. Both are in prospect.

Indications are this added capacity in Europe will be of skimming type, which would permit tops to be brought to this country for processing.

The people of Europe, despite their economic status, must be kept warm, and oil, once considered the luxury fuel, gives highest promise of accomplishment.

Progress Report

IN THE annual report of the Secretary of the Interior to the Congress, Dr. R. R. Sayers, director of the Bureau of Mines, gave this estimate of synthetic fuel progress:

"At the present rate of research progress, production of synthetic petroleum products on a commercial basis is possible within a few years."

Currently the report gives the cost range of fuel from coal or shale at "from 7½ to 9½ cents per gallon . . . not including profit or interest on the investment."

But the experimental laboratories set up in the research and development program, scientific progress has been sufficient to offer assurance of still lower costs. Following are quotations from a digest of the annual report, as released through the Information Service of the Department of Interior:

"In the hydrogenation process, lower pressures and lighter equipment are among the keys to production economy. Laboratory and pilot-plant studies have shown that coal may be converted to a distillable oil at relatively low pressures by employing special solvents and very active catalysts. Another radical departure from European practice is the discovery that coal may be hydrogenated in a dry or powder state, simplifying the process by eliminating several costly and complicated steps.

"In the gas-synthesis process, heat removal has been a major European problem, requiring expensive construction and limiting reaction-chamber output. A new type chamber controls temperature by evaporating a cooling oil over a catalyst. Production rates more than double those of European plants have been tested successfully and, even more important, this cooling method will permit enlarging the chamber to any size practical from a construction standpoint."

As to shale-oil development:

"Using a thermal solution process, which employs hot solvents to extract oil from shale, scientists have obtained oil recoveries greater than the assay value of the shale. Several engineering and technical obstacles remain to be overcome, however. Thermal-cracking studies on shale-oil fractions are nearing completion, and investigations of catalytic treating methods are under way. Considerable progress has been made in determining the constituents of oil shales and the properties of the shale oils. New methods have been developed for analyzing crude shale oils and determining hydrocarbon types in shale-oil naphthas. Other studies are in progress to determine the by-product potentialities of oil shale."

Good Job To Be Done

FOR all that private enterprise has done in providing mankind with goods and services, man in the mass continues to desert this scheme of economy and enter into collective ventures. It may be that man is simply stupid. Or it may be that industry has yet to present its free enterprise in attractive style. It does not seem reasonable for men to win a war or be fed by the products of capitalism, then turn to nationalization. Reasonable or unreasonable, England has a nationalized coal industry and a shortage of coal. Elsewhere the collective venture is expressed by standing in line to get a share of insufficient merchandise.

Perhaps this is the major task for good public relations. Properly told, the story of what free enterprise has done for mankind should win converts.

The Way To Cut

SO LONG as legislators try to designate the instances of reduced spending in government, there will be scant reduction in spending. Any head of a department can give convincing argument to justify his anticipated expenditure. Consequently a congressional committee simply plays one essential against another.

What the legislators should do is to leave ways of lower costs to the heads of departments. Just give the departments less money and the reduction in expenditure will follow.

Offices and employes are scattered over the whole nation. They occupy more office space and have more employes than before the war. A glance at what a department spent 10 years ago will give any committee a spot for setting up a flag of less spending, which will result in lower taxes.

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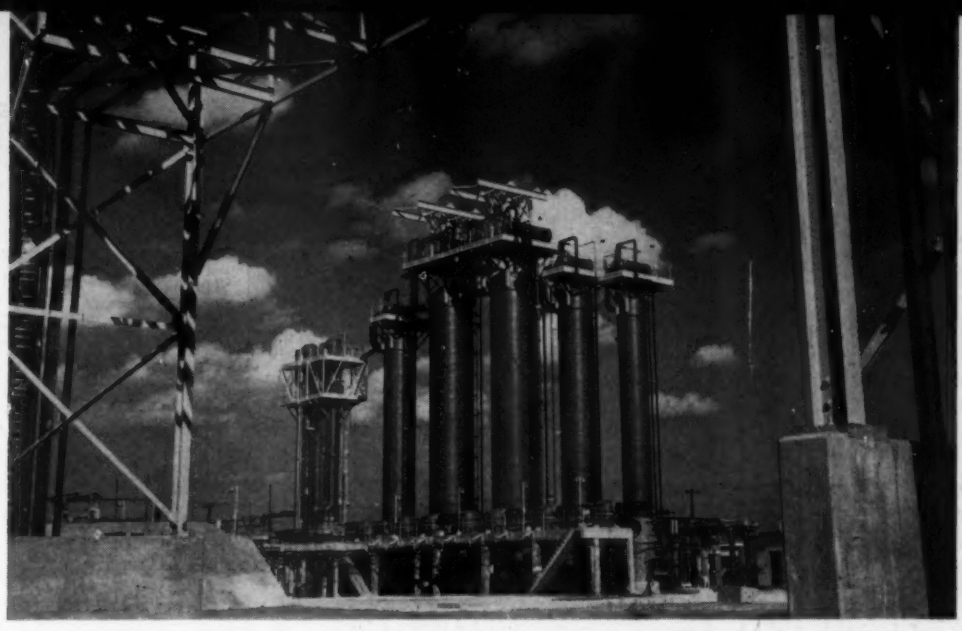

Fractionator bank for finishing styrene. Dow Chemical Company, Velasco, Texas.

Distillation Properties of Aromatic Hydrocarbons

Vapor-Pressure Chart, Improved Critical-Temperature and Latent-Heat Equations

JOHN GRISWOLD
The University of Texas
J. A. MAY
Dow Chemical Company, Styrene Division

A COX-TYPE vapor pressure chart for C_6 to C_{10} alkylbenzenes is developed from the recent Bureau of Standards tables and earlier critical constants.

The constants in Meissner's equation for critical temperatures of halogen- and sulfur-free compounds are evaluated from hydrocarbon data, resulting in an improvement of its accuracy. The equation with the new constants is,

$$T_c = 1.374T_b + 76 - r\,(0.325T_b - 74)$$

The Kistiakowsky latent heat equation gives improved accuracy for alkylbenzenes with the constants,

$$(ML/T)_b = 8.0 + 5.0 \log T_b$$

THE manufacture of products such as benzene, toluene, xylenes, styrene, cumene, phenol, and phthalic anyhydride entails fractionation of aromatic hydrocarbons.

Accurate boiling points, vapor pressures (in the low range), and latent heats of vaporization lately have been released.[1] These furnish a basis for a vapor-pressure chart and a re-examination of correlations of some of the properties needed for distillation calculations.

A number of hydrocarbon vapor-pressure charts have been developed on coordinate scales of functions of log P and reciprocal temperature. The most straight-forward method appears to be that of Cox,[3] modified by Calingaert and Davis.[2] The latter authors noted that the coordinates conformed to the Antoine equation,

$$\log P = A - B/(T + C) \qquad (1)$$

where T is °C., and C has the *average* value of 230 for normally-liquid hydrocarbons.

Recent work on vapor-pressure equations has shown that each compound has its own unique Antoine equation constants.[1] Furthermore, it is seldom that a single set of constants accurately represents vapor-pressure data of a compound over the wide range from subatmospheric to the critical pressure.[3,4] It follows that there is no single value of C that can be used in a chart temperature scale to provide straight lines for a variety of compounds over the desired wide range.

Apparently there are high-range vapor-pressure data only for the aromatics, benzene, toluene, and biphenyl. From unpublished work in this laboratory and scattered information in the literature, vapor-pressure data of a number of hydrocarbons lie close to and usually intersect a straight line drawn between the normal boiling point and the critical, on the coordinates of log P and 1/T, where T is an absolute temperature. This corresponds to a value of C = 273, with T in degrees centigrade. While the average value of C for alkylbenzenes is about 215 for the subatmospheric pressure range, the higher value is more suitable and near the optimum for the high range according to what information is now available.

The vapor-pressure chart (Figure 1) was constructed on the coordinates of log P and 1/T by plotting Bureau of Standards[1] data from 100 mm. to 1 atm., and drawing straight lines between the normal boiling points and the criticals. The existing high range data for benzene[7] and for toluene[9] are shown on the chart. The fit of the benzene data is satisfactory and better than the fit of the toluene data. The

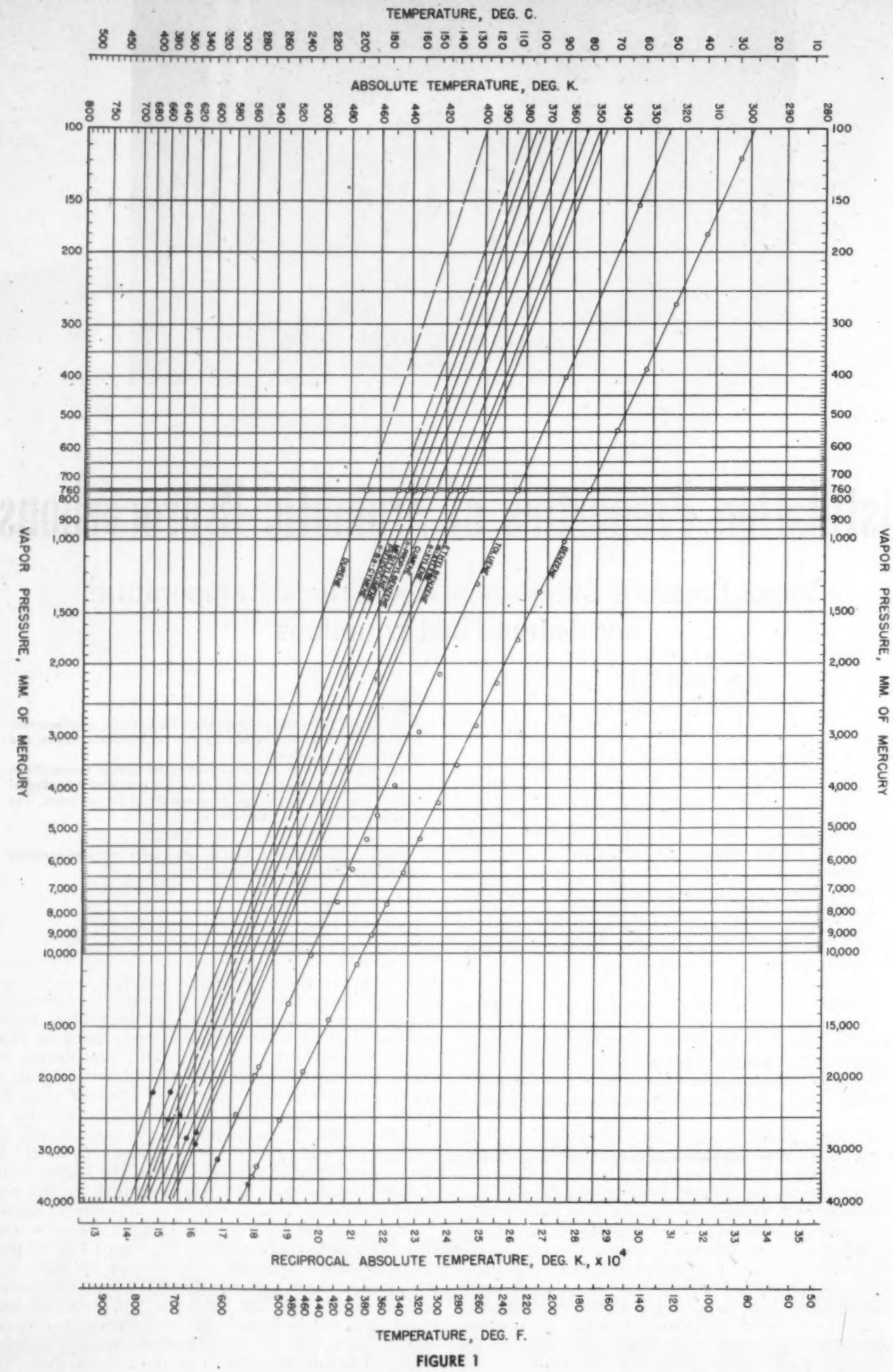
TEMPERATURE, DEG. C.
ABSOLUTE TEMPERATURE, DEG. K.
VAPOR PRESSURE, MM. OF MERCURY
RECIPROCAL ABSOLUTE TEMPERATURE, DEG. K., x 10⁴
TEMPERATURE, DEG. F.

FIGURE 1

TABLE 1

Comparison of Equations 2 and 3 for Critical Temperatures of Alkylbenzenes

	Literature	Calculated	
		Equation 2	Equation 3
Benzene	288.5 °C.	290.9 °C.	288.5 °C.
Toluene	320.6	326.2	323.2
Ethylbenzene	346.0	354.0	350.9
o-Xylene	358.3	364.8	361.4
m-Xylene	345.7	357.9	354.6
p-Xylene	344.4	356.9	353.7
Styrene	....	365.9	362.5
n-Propylbenzene	365.6	378.2	375.1
Cumene	362.7	369.6	366.4
Indene	....	408.0	404.5
Propenylbenzene	....	399.8	396.4
Mesitylene	367.0	385.3	382.0
Hemimellitene	....	400.0	396.6
Pseudocumene	381.2	391.2	387.9
o-Ethyltoluene	....	385.9	382.7
m-Ethyltoluene	....	380.9	377.8
p-Ethyltoluene	....	381.9	378.8
o & p-Cymene	378.6	393.3	390.3
Isobutylbenzene	377.0	387.0	384.1
Durene	402.5	419.6	416.3

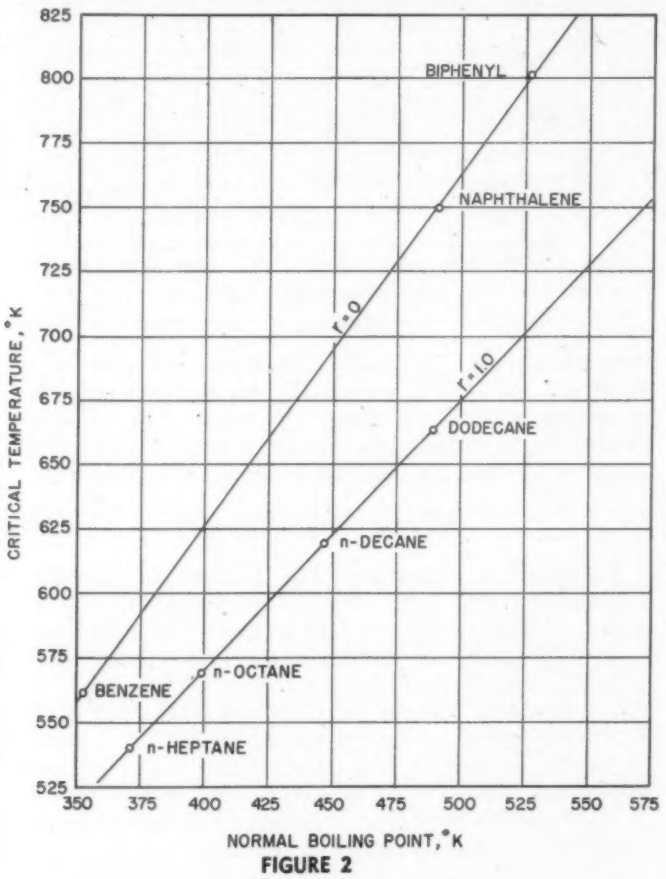

FIGURE 2

accuracy for higher aromatics should be of the same order. It is estimated that the curves represent the unknown vapor pressures to about 1.5 percent of the absolute pressure in the high range. Since the relative spacing of the curves at the normal boiling points and at the criticals is about the same, none of the curves cross and the values must be interconsistent. Such deviations as are present largely offset each other when the vapor pressures of the different compounds *are used as ratios,* as required for column-plate calculations. In view of the above considerations, some of the compounds whose boiling points are very close to others are omitted so as to give a clearer, more easily usable chart. Figure 1 is recommended as a good estimation of the nonexistent high-range vapor-pressure data.

Critical Constants

While the critical points on Figure 1 appear to be of reasonable accuracy, the majority were determined more than 50 years ago.[4] Means were sought to evaluate the accuracy or interconsistency of these data. While the attempt was but partially successful, it resulted in improved constants in the Meissner equation for predicting the critical temperature and in the Kistiakowsky equation for predicting latent heats of vaporization.

Several correlations of critical temperatures and pressures in terms of properties related to molecular structure have appeared. Herzog[4] gave a comprehensive survey of these to 1944, and presented equations of improved reliability for compounds in general.

Critical Temperatures

Meissner and Redding[11] presented the equation for prediction of critical temperature of halogen- and sulfur-free aromatics and naphthenes,

$$T_c = 1.41T_b + 66 - r\,(0.383T_b - 93) \qquad (2)$$

where T_b and T_c are normal boiling point and critical temperature (°K) respectively, and r is the ratio of noncyclic to total carbon atoms in the molecule.

This equation should be better adapted to alkylbenzenes than others not having the r-term. Assuming that the constants are unrelated, data on a minimum of 4 compounds are required to evaluate them.

So that no compound with an irregular relation would be included, critical temperatures were plotted against normal boiling points for inspection. The pure ring compounds (r = 0) benzene, naphthalene and biphenyl fell on a straight line, and the n-paraffins (r = 1.0) heptane, octane, decane and dodecane formed another straight line (Figure 2). Compounds having intermediate values of r fell between these lines but showed no conclusive regularities of position. With the straight-line relations of Figure 2, solution for the constants of Equation 2 gives,

$$T_c = 1.374\,T_b + 76 - r\,(0.325\,T_b - 74) \qquad (3)$$

Values calculated by Equations 2 and 3 are given in Table 1. Although both equations predict high values for alkylbenzenes, the values from Equation 3 are uniformly about 3° C. lower than the values from Equation 2. While a set of constants undoubtedly can be found to more closely represent existing data on alkylbenzenes it is not deemed advisable to attempt a closer fit until additional modern determinations are available.

Critical temperatures of the 21 additional halogen- and sulfur-free liquid compounds listed by Meissner were calculated by Equation 3 and are given in

TABLE 2

Comparison of Equations 2 and 3 for Critical Temperatures of Halogen- and Sulfur-Free Liquids

	MEISSNERS' TABLE I				
	Literature	Equations 5C & 5D		Equation 3	
			Difference		Difference
Acetone	508.2 °K	497 °K	−11.2	495.6 °K	−12.6
Acetic Acid	594.8	561	−33.8	560.6	−34.2
Isoamylformate	576.2	582	+ 5.8	582.7	+ 6.5
Aniline	699.2	709	+ 9.8	704.2	+ 5.0
n-Butyric Acid	628.2	607	−21.2	608.2	−20.1
Cyclohexane	554.2	564	+ 9.8	562.4	+ 8.2
n-Decane	619.4	618	− 1.4	619.4	0
Dodecane	663.7	661	− 2.7	663.7	0
Ethyl alcohol	516.3	519	+ 2.7	518.7	+ 2.4
Triethylamine	535.2	532	− 3.2	530.8	− 4.4
Ethylbutyrate	566.2	563	− 3.2	564.2	− 2.0
Ethylpropylether	500.6	502	+ 1.4	501.3	+ 0.7
1-Methylisopropylbenzene	651	665	+14	663.9	+12.9
3-Methyl-1-butene	464.5	460	− 4.5	457.9	− 6.6
Methyl alcohol	513.2	505	− 8.2	504.5	− 8.7
Naphthalene	749.7	759	+ 9.3	750.9	+ 1.2
Octadecane	763.7	769	+ 5.3	774.0	+10.3
Biphenyl	801.2	810	+ 8.8	800.4	− 0.8
Diphenylmethane	802.2	810	+ 7.8	802.7	+ 0.5
Phenol	692.2	708	+15.8	701.5	+ 9.3
Trimethylbenzene (Durene)	675.5	692.7[a]	+17.2	689.4	+13.9

[a] Reported as 682° in original reference.

Table 2. Although a number of the literature values are undoubtedly inaccurate, it is apparent that Equation 3 represents a marked improvement for hydrocarbons, a substantial improvement for benzene derivatives, and about the same accuracy as Meissner's equations for other types of halogen- and sulfur-free liquid compounds.

Critical Pressures

Several correlations have appeared that have not yet been compared for predicting critical pressures of alkylbenzenes. Among the most recent is one by Meissner and Redding[11] in terms of critical temperature and critical volume, and one by Gamson and Watson[5] for hydrocarbons only, by comparison with the critical pressures of n-paraffins having the same number of carbon atoms. Although they have not been recommended for this purpose, certain correlations based on corresponding states include and may be solved for critical pressure when everything else is known. For example, Othmer[12] gave the equation for correlating latent heats and vapor pressures, which may be written

$$\log P_r = (\log P'_r)\left[\frac{(ML)T'_c}{(ML)'T_c}\right] \qquad (4)$$

where P_r is reduced pressure, T_c is critical temperature (°K or °R), ML is molar latent heat of vaporization (at the same reduced temperature for both compounds), and the primed quantities are for a reference substance that may be water.

Equation 4 is solved readily for critical pressure ($P_c = P/P_r$) for any compound, knowing only the critical temperature and the latent heat at the normal boiling point.

Critical pressures for alkylbenzenes calculated by the three correlations above are given in Table 3.

Watson's correlation is the most accurate for this series of compounds although it does not account for the position and configuration of alkyl groups on the benzene ring. Othmer's correlation gives values up to several percent high, but within the general accuracy of correlations based on corresponding states.

Latent Heats

The Kistiakowsky equation,

$$(ML/T)_b = 8.75 + 4.571 \log T_b \qquad (5)$$

where T is in °Kelvin,

predicts molar latent heat at the normal boiling point of a wide variety of nonpolar liquids with an accuracy usually within 3 percent.[8] This equation was obtained from,

$$ML = V \ln V$$

where V is molar volume at 1 atmosphere, by substituting RT_b for V.

TABLE 3

Comparison of Critical Pressure Correlations for Values of Alkylbenzenes (Pressures in mm Hg)

	Literature	CALCULATED		
		Meissner	Watson	Othmer
Benzene	36,252	33,600	36,700	37,160
Toluene	31,616	29,320	31,210	33,630
Ethylbenzene	28,956	25,890	27,310	30,800
o-Xylene	28,044	26,400	27,310	31,020
m-Xylene	27,208	26,060	27,310	29,720
p-Xylene	26,600	26,010	27,310	29,660
Styrene	.....	27,600	27,310	.....
n-Propylbenzene	24,548	23,180	24,320	27,870
Cumene	24,472	22,860	24,320	29,390
Indene	.....	27,860	.....	.....
Propenylbenzene	.....	24,910	24,320	.....
Mesitylene	25,232	23,410	24,320	26,470
Hemimellitene	.....	23,940	24,320	.....
Pseudocumene	25,232	23,630	24,320	28,270
o-Ethyltoluene	.....	23,470	24,320	.....
m-Ethyltoluene	.....	23,280	24,320	.....
p-Ethyltoluene	.....	23,370	24,320	.....
o & p-Cymene	21,736	20,620	22,250	25,220
Isobutylbenzene	23,637	20,430	22,250	24,130
Durene	21,736	21,540	22,250	25,120

If the constants of Equation 5 are regarded as empirical and are evaluated from experimental data, a considerable improvement in accuracy is obtained. The

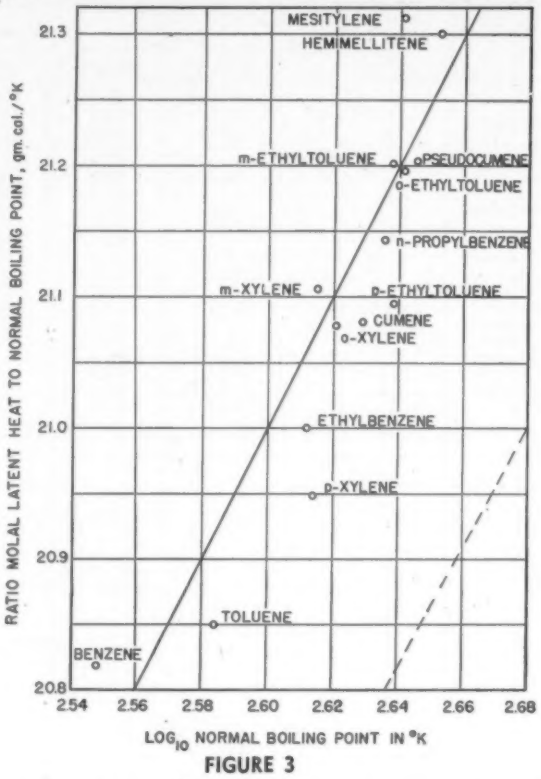

FIGURE 3

latent-heat data for alkylbenzenes are plotted on Figure 3 as ML/T versus T_b, in conformance with Equation 5 which is the dashed line. The solid line drawn so as to include benzene and toluene has a slope of 5.0 and an intercept of 8.0 giving,

$$(ML/T)_b = 8.0 + 5.0 \log T_b \qquad (6)$$

Equation 6 reproduces the Bureau of Standards values for latent heats of alkylbenzenes with discrepancies ranging from +0.59 percent for p-xylene to −0.49 percent for mesitylene. It is generally less accurate than Equation 5 for hydrocarbons other than alkylbenzenes. Latent heats at pressures other than 1 atmosphere may be estimated to within 5 percent or better by any of the correlations based on corresponding states that involve these properties.[10, 12, 13, 15] Watson's equation in terms of reduced temperature only is particularly convenient,

$$L/L_1 = \left[\frac{1 - T_r}{1 - T_{r1}}\right]^{0.38} \qquad (7)$$

where L and T_r are taken at one temperature, and L_1 and T_{r1} are taken at another temperature.

Equation 6 may be used to obtain one of the latent heat values.

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Custom Building Lubricants With Additives

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A FUNDAMENTAL scientific approach to the variables involved in lubrication has been followed in the physical laboratories, where the effort still continues vigorously. Necessity for a day-to-day series of practical lubricants has led to experimental study, along much different lines, of how to make lubricants work. In this paper an attempt is made, using crankcase and hypoid-gear lubricants as examples, to show some of the techniques recently used employing the immediately practical rather than the fundamental method of approach to secure compounded lubricants of value in the field.

The text of this paper is similar to material presented by the author in a talk before the Texas-Louisiana-Gulf section of the American Chemical Society in Port Arthur recently, and the illustrations have been made from slides shown at that meeting.

THE art of compounding lubricants is undoubtedly old, very probably dating back to the first rudimentary wheels and axles when ingredients were blended with the proper incantations—a practice which has not wholly fallen into disuse. The *science* of compounding lubricants with additives is relatively new, and the body of knowledge affecting it is still far from complete.

The essential problem in lubrication is one of finding a lubricant permitting the most economic functioning of a mechanical device; i.e., maximum efficiency and minimum maintenance. So far there have been two major approaches to the broad general problem of lubrication and the many special problems.

There has been the fundamental scientific attack which seeks to define the variables involved in lubrication, to isolate each of these variables, and to study means of affecting the individual variables. This method has been followed primarily in the physical laboratories where the attempt still continues vigorously to define the functions of a lubricant and study the effect of the variables on such functions.

The second method of attack is based on the necessity of having a day-to-day series of practical lubricants available for use. The methods of this type of attack have been dictated by the problems arising in the field, and have largely consisted of attempts to find means of simulating field conditions on a reasonably small scale. In this method of attack, attempt is made, not to study each variable in the absence of all others, but to control all variables at a constant level and then change one to determine its effect. This type of attack is, therefore, basically more complex and less easy to interpret than a fundamental physical study of the properties of lubricants. The absolute control of all the variables is difficult, if not impossible, in dealing with complicated machinery. Furthermore, the problem of defining field conditions is major, and must be faced in each case, because field-service record is the final criterion. In this paper an attempt will be made, using crankcase and hypoid-gear lubricants as an example, to show some of the techniques recently used employing the "immediately practical" rather than the fundamental method of approach to secure compounded lubricants of value in the field. Particular emphasis is to be placed on differences in performance encountered in treating a wide variety of base stocks.

Practical Study

In the "practical" study of how to make lubricants work, several functions have been isolated. To name a few:

1. Ability to flow at low temperature.
2. Resistance to deterioration.
3. Resistance to the tendency to corrode hard-alloy bearings in the case of crankcase oils.
4. Resistance to ring-zone deposits and engine deposits generally in the case of crankcase oils.
5. Resistance to extreme pressure conditions.
6. Resistance to wear.

All of these functions must be performed without loss of intrinsic lubricating ability. To meet these conditions the modern compounder of lubricants has available chemical materials developed to improve each one of these properties. He, generically, has available a wide selection of base stocks arising from different sources of crude and different techniques of refining. With these a wide variety of finished lubricants may be prepared. In the particular blender's case, however, the variety of base stocks from which selection may be made is usually somewhat restricted.

In order to secure lubricants of practical economic value by the proper interpretation of test results, it must be borne in mind, however, that the use of additives and their performance is not by any means independent of base stocks.

Different base stocks arising from different crudes and processed by different refinery techniques would, of course, be expected to show some difference in performance and additive response. Response, however, may be quite critical even with relatively minor refining differences if the lubricant sought is the most economic formulation. It has been found impossible to generalize on the performance of an additive from one base stock to another except within certain limits. It is possible to say that if the usually recommended dosage of additive is employed certain levels of performance will be met. These barely may be met or exceeded. To determine the proper balance for a given base stock requires investigations that increases in cost with precision. The amount of information collected in a given case, therefore, must be balanced economically.

Cold-Flow Property of Oils

As a first case in the survey of "practical" approach to lubricant function and the compounding of lubricants, let us consider the cold-flow property of oils.

Field results have shown modern oil sometimes thickens or solidifies at impractically and embarrassingly high temperatures. Control of this property, aside from the viscosity effect, can be exercised by selection of crude and by vigorous dewaxing. This, however, is expensive and the individual blender having available only certain base stocks, must make them work. This can be accomplished conveniently and economically by means of pour-point-depressant additives.

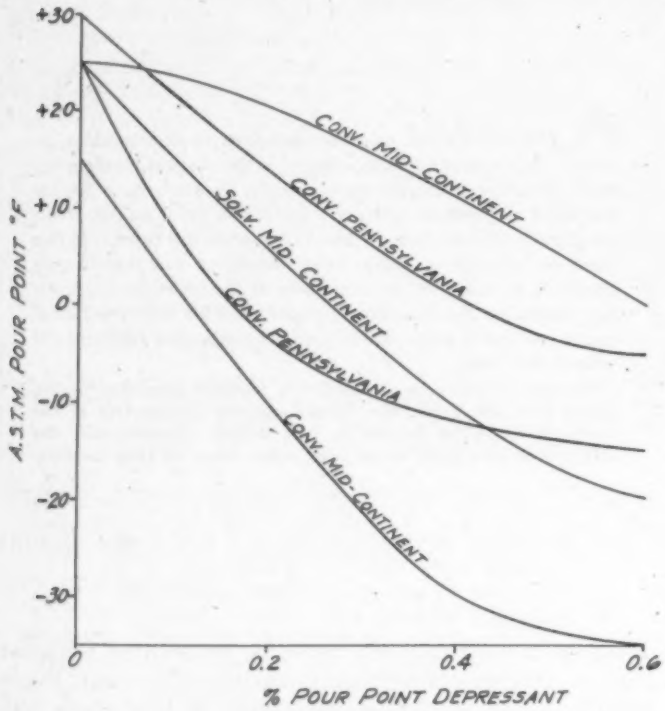

FIGURE 1

Variation in pour-point-depressant with base stock.

Many laboratories have engaged and are engaged in evaluating the cold-flow properties of compounded oils. Several methods of evaluation have been proposed for which varying degrees of correlation with field results have been claimed. If we take the ASTM pour-point method as exemplary of these (although this, admittedly, does not give a complete answer on the field stability of compounded oils), we can examine variation in base stock response.

Figure 1 shows a plot of ASTM pour points vs. pour-point-depressant concentration for several base stocks. It can be seen that, if sufficient pour-point depressant is used, almost all oils can be brought to a satisfactory ASTM pour-point, but the same concentration may produce widely different pour-points in different base stocks. Each of the proposed laboratory methods of evaluating pour-point-depressant response shows variability from base stock to base stock as do field results themselves.

Since the existing laboratory evaluation methods have either been criticized as to validity or are as yet not sufficiently substantiated by field data secured over a long period, heavy dependence must be placed on field results.

In the case of compounding lubricants with pour-point depressants, therefore, broad generalization on additive performance is limited by both testing technique and base-stock response variation. However, experience has shown it possible, on the basis of extensive field testing and laboratory work, to recommend concentrations of additives for groups of base stocks with reasonable assurance of meeting certain minimum requirements. The optimum point, or point of ultimate economic balance, is obtainable, however, only by extensive testing of a give additive—base-stock combination.

Prevention of Deterioration

The problem of prevention of deterioration of oil in an engine and the prevention of bearing corrosion is old. Much can be done towards minimizing the problem by proper selection of crudes and refinery techniques. However, ultimately the blender is confronted with the problem of making the finished stocks available to him work satisfactorily. This can be done usually only with an inhibitor. Field experience has broadly defined the problem, and field tests still represent the final evaluation. However, many laboratory and engine tests have been devised to evaluate corrosiveness and stability of oils without the expense of elaborate field trials. Correlation of these tests with field performance has been of varied success. During the recent war the United States Army Ordnance Department was confronted with the problem of selecting suitably non-corrosive and stable oils on a truly massive scale. Through the cooperative effort of the CRC, (Coordinating Research Council) the 36-hour Chevrolet test (CRC L-4-545) finally was selected as the best available tool for such evaluation.

FIGURE 2

Bearings from L-4-545 Chevrolet tests. Left: Straight oil, no additive; Center: Base oil A with 0.75 percent inhibitor X; Right: Base oil B with 0.75 percent inhibitor X.

Qualification under Army Specification 2-104A and 2-104B was based partly upon this test.

There may be some comment that meeting the requirements of the above specification may be too severe a test for ordinary civilian use. However, it should be noted that, in Army experience, oils satisfactorily meeting the above requirements were uniformly satisfactory in the field. Any error inherent in this criterion was, therefore, on the conservative side,

The resistance to deposition of sludge and lacquer, particularly in the ring zone, in both spark- and compression-ignition engines is an important criterion of long operation between overhauls and of efficiency of operation. The ability of an oil to keep products of oil deterioration in suspension, promoting engine cleanliness, commonly is defined as the "detergency" or "dispersancy" property of the oil. Again, careful selection of crudes and refining techniques can pro-

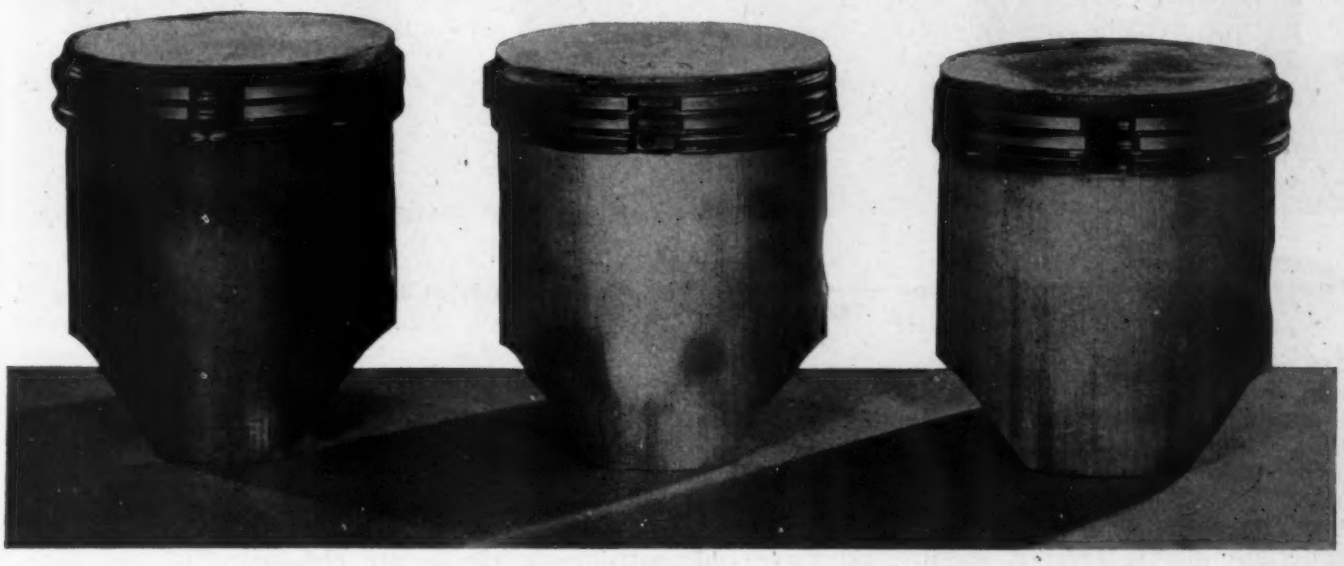

FIGURE 3

Pistons from L-4-545 Chevrolet tests. Left: Straight oil; Center: Base oil A with 0.75 percent inhibitor X Right: Base oil B with 0.75 percent inhibitor X.

and additive oil satisfactorily passing the Chevrolet test can be considered to be of minimal corrosiveness and good stability.

Passing to the results of some of these tests, Figure 2 shows three copper-lead bearings from Chevrolet tests. The darkest one of the bearings is from a test run on a good base oil containing no inhibitor and showed excessive weight loss. The other two bearings are from tests on two different base oils containing the same amount of inhibitor. The middle bearing showed acceptable weight loss within the normal wear range. The slight discoloration is interpreted to be indicative of minimum passable inhibitor concentration. By contrast, the third bearing, while showing no significantly lower weight loss, is completely free of discoloration retaining its "new bearing appearance" and indicating outstanding quality.

Roughly the same gradations in quality are encountered in examination of varnish deposits on pistons used in the Chevrolet L-4-545 test. Figure 3 again shows a blank oil compared with two different base stocks containing the same inhibitor concentration. The center piston would be considered a minimal pass for a present premium oil. The one on the right, is, of course, an outstanding oil.

The variation in performance from base stock to base stock again limits generalization. However, an inhibitor capable of showing marked improvement in bearing corrosion and stability in some stocks will always show some improvement even in the most refractory bases. Moreover, again it is possible to set recommended concentrations for groups of base stocks which assure at least minimum performance The exact level of performance desired and securable as well as the economic balance are still the subjects for considerable testing of a given base stock-additive combination.

duce relatively clean-running straight oils. However, abundant field data have shown that for heavy-duty performance additive-compounded oils are required, and for medium-duty performance, are desirable.

In evaluation of "detergency," also, the war experience of the Army and the cooperative work of CRC, led to the selection of the Caterpillar 500-hour test, CRC L-1-545, as a criterion. Again broad military experience showed oils passing this test to have satisfactory detergency properties in the field. The exact amount of such "detergency" required in civilian vehicles is being determined by service study in the field with the L-1 test remaining as the major criterion of such performance short of the actual field tests themselves.

Figure 4 shows two pistons from Caterpillar L-1-

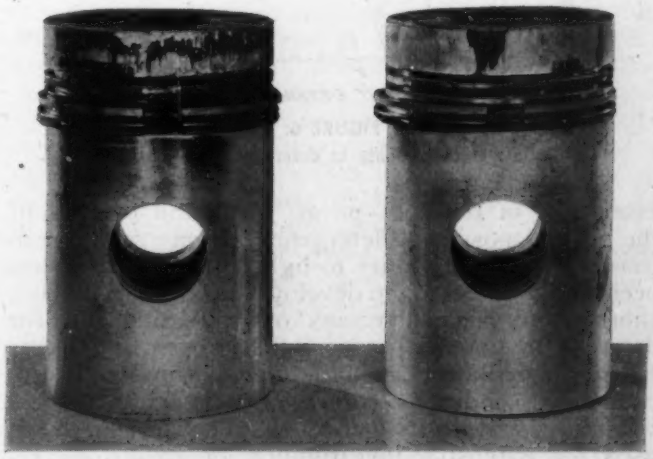

FIGURE 4

Pistons from L-1-545 Caterpillar tests. Left: Base oil C with 3.25 percent additive Y; Right: Base oil D with 3.25 percent additive Y.

FIGURE 5

Pistons from Lauson detergency tests. Left: Poorer reference oil—rating 90; Center: Intermediate oil (borderline 2-104B detergency)—rating 92; Right: Uppr reference oil—rating 94.

545 test run with the same additive concentration in two different base stocks. One represents a borderline failure for 2-104B performance on piston-skirt lacquer and ring-groove carbon. The other represents a clear pass. This again illustrates variability of base stock response and gives rise to the usual problem of how to determine the optimum.

Because the L-1 test is lengthy, expensive and

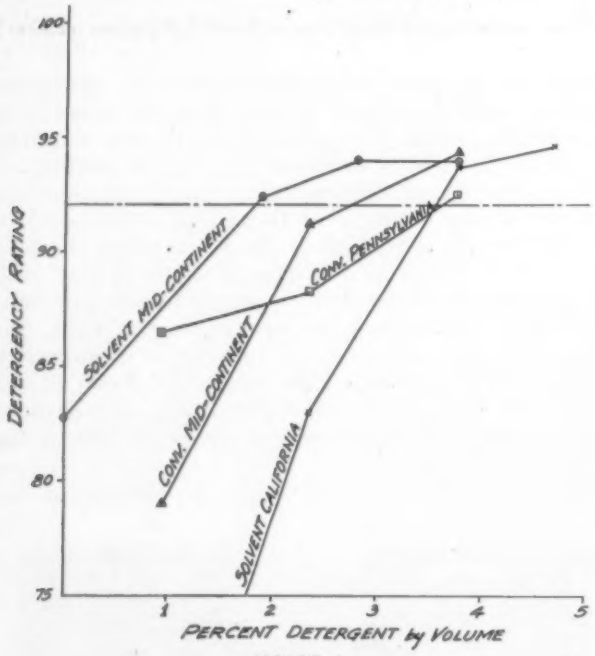

FIGURE 6

Base-stock response to detergent treatment.

essentially of the "go—no go" type and because of the several possible "detergency" levels required in passing from heavy-duty to light-duty service, it has been found necessary to develop a reasonably precise, shorter and cheaper means of evaluating oils for detergency. For this purpose it has been found that the single-cylinder Lauson engine run under strictly controlled conditions correlates well with the L-1 test and therefore with the field. This test has been used for extensive investigation of base-stock response to detergents.

It is worth noting, in passing, that this Lauson test differs considerably from those previously reported. Extreme precision in control of dimensional tolerances and operating temperatures is required. Each series of three test oils in each engine is bracketed between two reference oils, one of outstanding L-1 performance and one of slightly less than passing 2-104B L-1 quality. As well, the same oil is run in at least two Lauson engines—or more—until satisfactory checks are obtained. The detergency rating so obtained is on a 0-100 scale with 100 representing a clean piston skirt.

Figure 5 shows three such Lauson pistons. One piston is from the poorer reference oil, one the better reference oil and one a test oil of about borderline 2-104B L-1 passing characteristics. Using this technique the data given in Figure 6 have been assembled on base-stock response.

In the case of detergents, as in the case of inhibitors, although satisfactory test techniques are

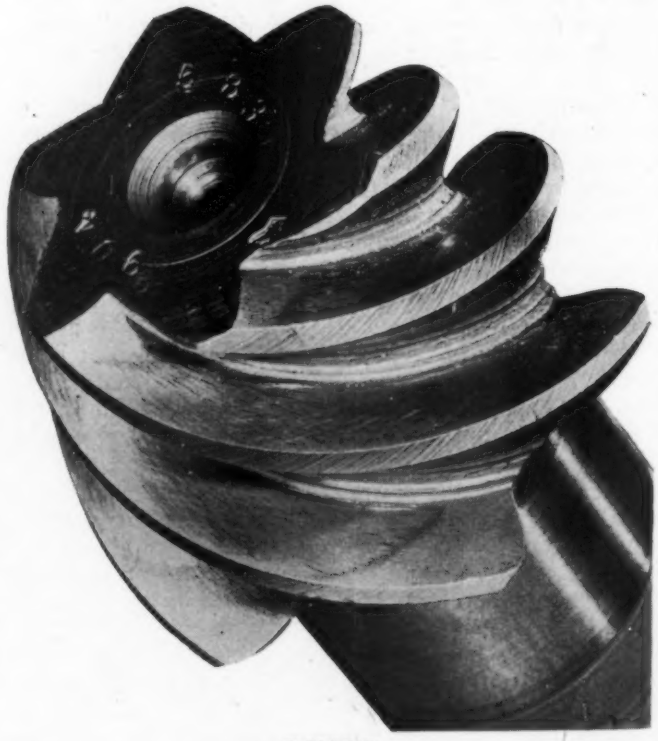

FIGURE 7

Pinion gear from L-20-545 axle test. Base oil E with 17.25 percent additive Z.

available, correlating well with the field, generalization on performance is limited by base-stock response. However, as can be seen, it is possible for a group of base stocks, to recommend dosages which will assure the meeting of minimum requirements for either medium-duty or heavy-duty service. The determination of the precise point of economic balance, aside from the problem of defining "medium duty," is dependent on the testing of a given additive-base stock combination.

Gear Lubricants

In the case of the compounding of gear lubricants, field requirements have imposed conditions of from mild oiliness to extreme pressure resistance on lubricants, depending on the type of gear and the nature of the service. Before the war much emphasis was placed on hypoid lubricants meeting U. S. Government VVL-761 Specifications for extreme-pressure lubricants. This was one case where variation in base stocks was of minor importance with several commercial additives available which, at their recommended dosages, enabled almost all base stocks to meet the deceleration test. This specification was taken over almost bodily into U. S. Army 2-105 and 2-105A Specifications with uniformly satisfactory results on lubricants so procured.

However, it was also realized that more emphasis might be placed on the wear-resisting properties and low speed-high torque performance of hypoid lubricants. To this end, by the cooperative effort of the CRC, the modified U. S. Army Specification 2-105B was developed and issued, including the CRC L-20-545 test for low speed-high torque characteristics.

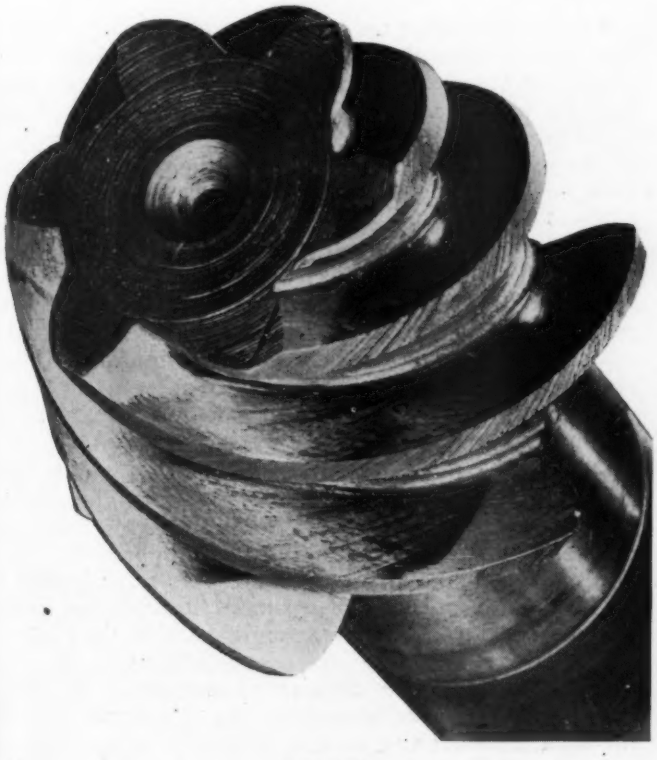

FIGURE 8
Pinion gear from L-20-545 axle test. Base oil F with 17.25 percent additive Z.

Figures 7 and 8 show two pinion gears from a CRC L-20-545 test on the same additive concentration in two different base stocks. One is a clear pass showing only a light polish. The other shows a definite "fish scale" or ripple and would fail. To illustrate a further criterion of failure on this test, Figure 9 shows a ridged pinion gear.

Once again base-stock response is shown to be variable and generalization limited. However, at the recommended dosages, a group of related base stocks show essentially the same performance and would be approved under Specification 2-105B. The ultimate economic balance again would require extensive testing of a given additive-base-stock combination.

FIGURE 9
Base oil G with additive H.

Summary

In the above outline a review has been made of some of the methods of evaluating crankcase and hypoid-gear lubricants with particular attention to requirements for matching additives and base stocks. It has been pointed out that the determination of the economic optimum additive dosage to meet a given performance level is complex even in those cases where the performance level is clearly defined in terms of readily accessible semi-large or laboratory tests. However desirable it might be to determine precisely the point of economic optimum additive dosage in each case, such a program requiring such extensive testing on a particular additive-base stock combination is very expensive and would contribute materially to the cost of the finished lubricant. This cost, therefore, must be balanced against the gain due to adjustment of dosage.

The examples cited above could be amplified by addition from many other fields of lubrication as well as by other additions from the field of crankcase and gear lubricants. In all cases, the problem of custom building lubricants with additives follows the same pattern:

1. Defining the functions to be modified.
2. Establishing the cost-performance level desired.
3. Establishing the economic additive dosage with the least cost of testing justified by the particular problem.

Simplified Charts for Petroleum Process Calculations

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IN THE study and design of process equipment, the chemical engineer has at his disposal three fundamental relationships. These consist of (1) material balances, (2) vapor-liquid equilibrium conditions, and (3) heat balances.

The material balance is self-evident and requires only that every quantity of material entering a unit must appear in some manner or form in the products leaving the unit. The other two relationships require a large number of charts of data in order to carry out the calculations on petroleum process equipment. While it is tedious and time-consuming to refer to all these charts for the necessary calculations, it is possible to keep such a data book in the office. It is, however, particularly inconvenient for an engineer in the field to continually carry a large cumbersome data book with him for these calculations. Thus, a method has been developed for representing all the necessary data for the vapor-liquid equilibrium and heat-balance calculations on a minimum of three charts. This has been done by representing the data as nomographs on which it is possible to represent a single curve by two lines and a point, a family of curves by three lines, or several families of curves by the appropriate number of lines.

Vapor-Liquid Equilibrium

The vapor-liquid relationship gives the vapor and liquid concentrations in equilibrium at different temperatures and pressures. With the exception of the hydrocarbons, these data on mixtures containing more than three components are virtually non-existent. The reason is that the mixtures of the hydrocarbons are very nearly ideal and the data can be represented by a simple relationship, whereas in the case of a non-ideal mixture of more than three components, the data cannot be represented in a simple manner.

In an ideal mixture, the partial pressure of any component is proportional to its concentration and independent of the composition of the balance of the mixture. This is known as Raoult's law and may be expressed as

$$p = P_o x$$

p = partial pressure of the component in the vapor.
x = mole fraction of the component in the liquid.

In this relationship, P_o is the proportionality constant, however, it can be seen that the partial pressure of the pure component, i.e., when $x = 1$, is equal to this proportionality constant. Thus P_o is the vapor pressure of the pure component at the given temperature.

By Dalton's law, $P = \Sigma p$, and by Avogadro's law it is possible to express the partial pressure of a component as the fraction of the total pressure in terms of the concentration in the vapor, and thus

$$y = \frac{P_o}{P} x$$

y = mole fraction of component in vapor
P = total pressure of vapor.

At a given temperature and pressure, the ratio of P_o/P is a constant and is known as a K-value.

$$y = Kx$$

The deviations from Raoult's law and the perfect-gas laws frequently are appreciable, particularly at pressures above atmospheric, and thus the K-value may be more accurately defined as the ratio of the fugacity of the liquid to the fugacity of the vapor.[5] In its application, however, it is considered to vary only with temperature and pressure.

The K-values of the different components generally are represented on separate graphs showing either the variation with temperature at different pressures or the variation with pressure at different temperatures.[1] Thus, in order to apply these K-values to the calculations on mixtures containing numerous different hydrocarbons, it is necessary to refer to as many different graphs. This is time-consuming even if all the necessary charts are at hand.

Recently a nomograph was developed which presents the K-value data for all the normal paraffin hydrocarbons from methane to tetradecane and also ethylene, propylene, isobutane and isopentane.[6] This nomograph is shown in Figure 1. A straight edge is placed on the nomograph to pass through the given temperature on the extreme-right-hand scale and the given pressure on the extreme-left-hand scale. The K-values for all the different components at these conditions can then be read on the appropriate intermediate scales. Thus, all the desired K-values can be determined immediately by a single setting on the nomograph.

The nomograph was developed empirically by calibrating the temperature scale according to logarithm of the liquid fugacity of n-pentane and calibrating the pressure scale according to the logarithm of the vapor fugacity of n-pentane. If the vapor fugacity were independent of temperature, the K-value scales would be straight lines and logarithmically calibrated. It is possible to compensate for the deviations by locating the component scales empirically, and the results are the curves shown in Figure 1. For tem-

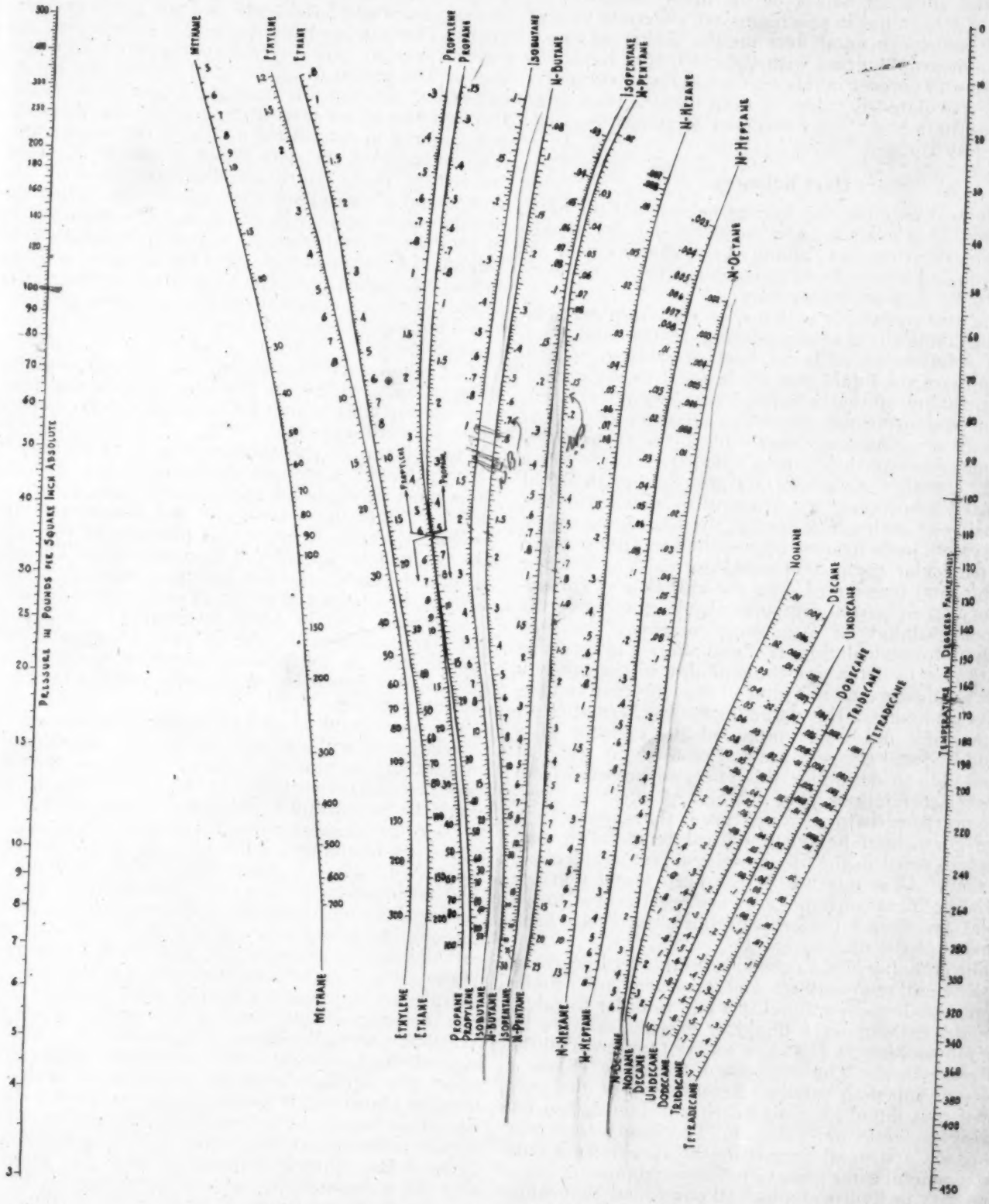

FIGURE 1
Nomograph for Equilibrium Constants of Hydrocarbons.

peratures up to 300° F. and pressures between 10 and 300 psia, the values given by the nomograph agree, within an accuracy of 2 percent, with the calculated K-values. Temperatures and pressures above these values are close to the critical region for most of the components, where the K-values vary with composition and chemical nature of the other components. Thus, the K-values in this region are uncertain unless based on experimental data on the actual mixture. The nomograph agrees with the calculated K-values to within 10 percent in this region but the uncertainty of the calculated K-values is even greater than this. The limitations of the calculated K-values are discussed by Dodge.[2]

Heat Balances

The heat entering any system must equal the heat leaving. This must include the heat contents of the streams entering and leaving the system as well as heat applied externally as a reboiler duty, or removed externally as a condenser duty.

The heat content or enthalpy of a mixture depends on the composition of the mixture. In the case of an ideal solution, there is no heat of mixing, so the enthalpy of the liquid mixture is equal to the sum of the enthalpy of the individual components at their given concentrations. Since the mixtures of hydrocarbons are considered nearly ideal, the enthalpy of a liquid mixture thus can be calculated.

The enthalpy of gaseous mixtures can be calculated similarly; however, an attempt to determine the enthalpy of each component at the total pressure on the system leads to some impossible conditions where the particular component could not exist as a vapor at the total pressure. Using the enthalpy of components at their partial pressures also does not give the proper enthalpy of a gaseous mixture. The best method for calculating the enthalpy of a gaseous mixture is to determine the enthalpy of the mixture at zero pressure as the total of the different components, and correct this value for the isothermal effect of pressure. Kay[4] has proposed the concept of a pseudo-critical temperature and pressure by which it is possible to determine this effect of pressure from general correlations. The isothermal effect of pressure on the enthalpy of a mixture is the same as for a pure component having a critical temperature and pressure equal to the pseudo-critical temperature and pressure of the mixture. It has been found that the pseudo-critical constants are very nearly equal to the molal average of the critical constants of the components of the mixture.

The enthalpy of the normal saturated hydrocarbons at different temperatures and pressures are usually represented on separate charts and in order to calculate the enthalpy of a liquid or gaseous mixture by the previous method, it is necessary to refer to all the different charts. The nomograph shown in Figure 2 was developed to present the enthalpy of the pure vapor and liquid on a single chart.[7] The datum enthalpy is liquid at —200° F. The nomograph was based on a general correlation of specific-heat data and a general correlation of pressure-volume-temperature data of hydrocarbons.[3] It was found that when the vapor enthalpy of a hydrocarbon was plotted as a straight line against an empirical temperature scale, the enthalpy of all other hydrocarbons were very nearly straight lines when plotted on the same scales. It also was found that the pressure was a linear function at a constant enthalpy when plotted against this empirical temperature scale. The nomograph was constructed by calibrating the temperature scale according to the previous empirical function and calibrating the pressure scale uniformly. The vapor-enthalpy scales were located empirically for all the different normal paraffin hydrocarbons.

Thus, when a straight edge is placed on the nomograph so it passes through the given temperature on the extreme-right-hand scale and the given pressure on the extreme-left-hand scale, the enthalpy of the pure vapors are given on the appropriate component scales. The enthalpy of the pure liquids can be obtained by placing the straight edge so it passes through the given temperature point and the focal point at the upper-left-hand side of the nomograph. The enthalpy of the pure liquid up to 50° F. below its critical temperature can be determined on the component scales above the dashed line. When the component is present in the liquid at within 50° F. of its critical temperature, or above, it can be considered as an absorbed gas, and the enthalpy is given below the dashed line. The heat of absorption varies with concentration and the enthalpy values given by these scales correspond to the variation encountered in practice.

The nomograph reproduces the original enthalpy charts almost within the precision of their construction and certainly within the limits of accuracy of the basic correlations.

The componential enthalpy made up as previously described is very tedious, and several such calculations must be made for each heat balance. It is possible to relate the enthalpy of the mixtures to the molecular weight because this property of the mixture is a definite function of its composition. The correlation is based on the normal saturated hydrocarbons, and since the mixtures encountered in practice usually contain some unsaturated compounds and some isoparaffins, this is only an approximate relationship. In the absence of more specific data, it is usually considered sufficiently reliable for engineering purposes.

The nomograph shown in Figure 3 represents the correlation of enthalpy with molecular weight of the mixture.[7] Inspection of Figure 2 indicates that the lines of constant enthalpy in the lower right corner would be practically vertical and almost parallel to the lines of constant molecular weight. Thus, in this region the enthalpy of the mixture would be very sensitive to the molecular weight and could not be read very precisely. The precision could be greatly improved by adjusting the datum enthalpies of the different hydrocarbons so the lines of constant enthalpy at the center of the figure will be straight. These datum enthalpies were chosen so they are additive for components heavier than propane. This was determined by plotting the enthalpy of the hydrocarbons at the datum temperature of —200° F. against the reciprocal of the molecular weight. The curve was straight over the range from octane to propane; however, it was necessary to allow a slight curvature for ethane and methane. The choice of any datum enthalpy would cancel in any heat balance because the material balance on the unit requires that the same quantity of components appear in the streams both entering and leaving the system, and the net heat effect is the difference between their enthalpies. The additive datum enthalpies may deviate slightly from the enthalpy of the mixture of the corresponding molecular weight given on the upper horizontal scale of Figure 3, however this is of minor significance because even if the enthalpies were

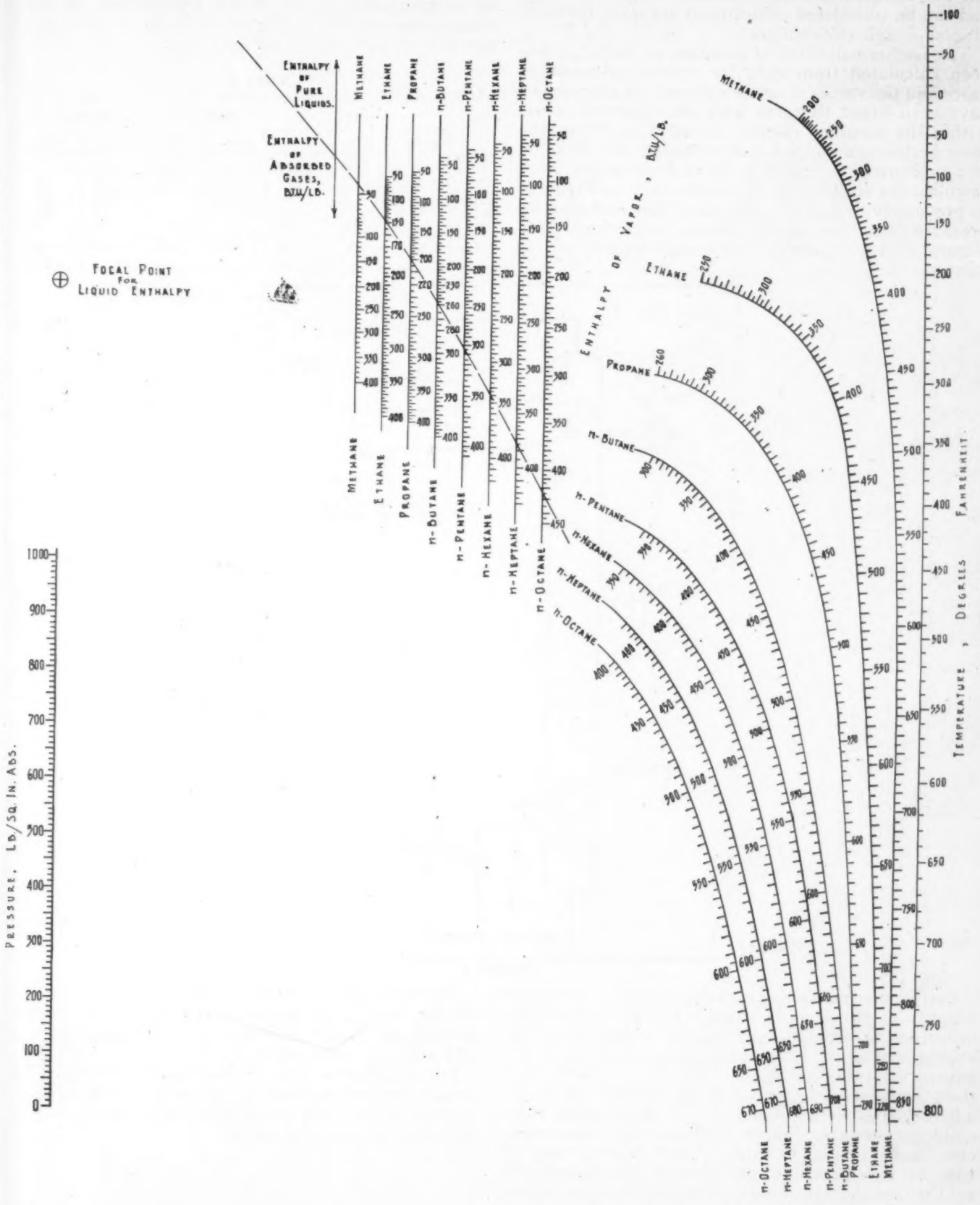

FIGURE 2
Enthalpy Nomograph for Pure Hydrocarbons.

chosen to be additive at —200° F., they would not necessarily be additive at other temperatures. Thus, the nomograph in Figure 3 is not theoretically exact[7] but the correlation with molecular weight is simple and can be considered of sufficient accuracy for most process-design calculations.

The isothermal effect of pressure on enthalpy has been calculated from data for several mixtures of gases and the values determined from the nomograph have been found to agree with the observed values within the accuracy of the original data. Thus, the most accurate method for determining the enthalpy of a mixture of saturated hydrocarbons would be to calculate the enthalpy at zero pressure from Figure 2, as previously described, and correct this enthalpy for pressure according to the values determined from Figure 3 at the average molecular weight of the mixture.

Application of the Nomographs to Process Calculations

The use of these nomographs can be demonstrated by carrying out a few of the calculations on the debutanizer shown in Figure 5. Table 1 gives the material balance on the column.

TABLE 1

	FEED			OVERHEAD		BOTTOMS	
	m/hr.	M.W.	#/hr.	m/hr.	#/hr.	m/hr.	#/hr.
C_3.....	1.0	44	44	1.0	44	.....	.....
C_4.....	40.0	58	2320	39.4	2285	.6	35
C_5.....	23.0	72	1656	.4	29	22.6	1627
C_6.....	16.0	86	1376	.....	.....	16.0	1376
C_7.....	12.0	100	1200	.....	.....	12.0	1200
C_8.....	8.0	114	910	.....	.....	8.0	910
	100.0		7506	40.8	2358	59.2	5148

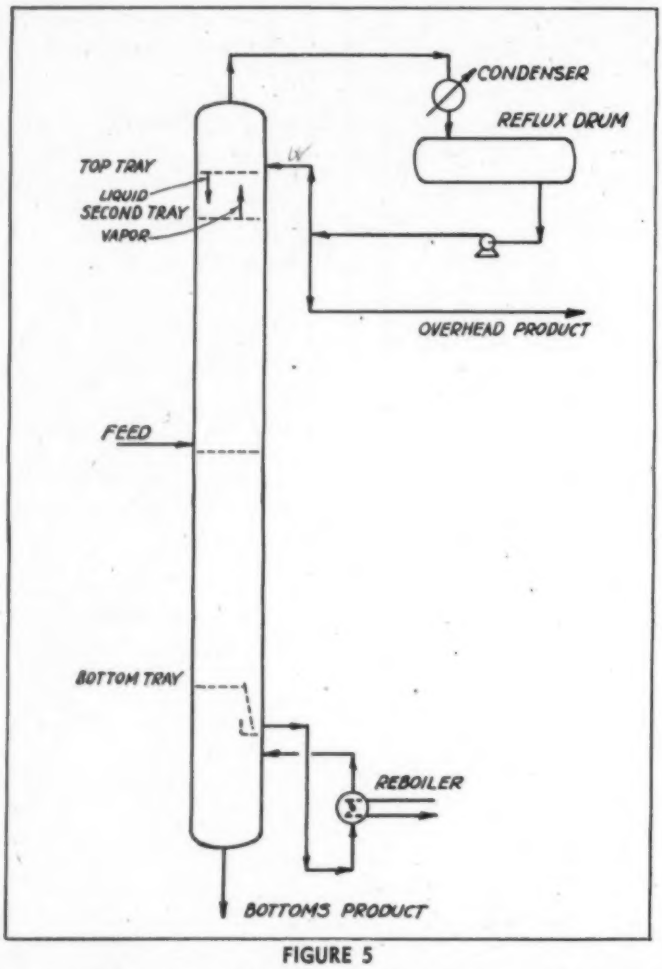

FIGURE 5

Petroleum mixtures having average molecular weights of 80 or more usually contain appreciable quantities of aromatic compounds which affect the average enthalpy. Thus, the correlation based on the normal saturated paraffins would not be reliable. For these higher-boiling mixtures, the specific gravity is a better basis for the correlation of the enthalpy than molecular weight. Figure 4 shows the nomograph constructed similar to Figure 3 but showing lines of constant API gravity of the mixture. The nomograph is based on the data for an oil having a characterization factor of 11.8. The nomograph would be slightly different for other oils, however, the data on other oils are not very complete. The previous oil represents an average of the different crude oils encountered and thus this chart may be used for general application without serious error.

The tower is to operate at 100 psia pressure and the the feed is to be preheated to 220° F. before entering the tower. The unit is to be designed for a cold external reflux ratio of 3:1.

The equilibrium flash on the feed is calculated by a trial-and-error method of assuming a ratio of L/V and calculating the quantity of each component in the vapor by the relationship:

$$v = \frac{f}{\frac{L}{VK} + 1}$$

where f = moles of a component in feed
K = K-value of component at given temperature and pressure
L = total moles of liquid
V = total moles of vapor

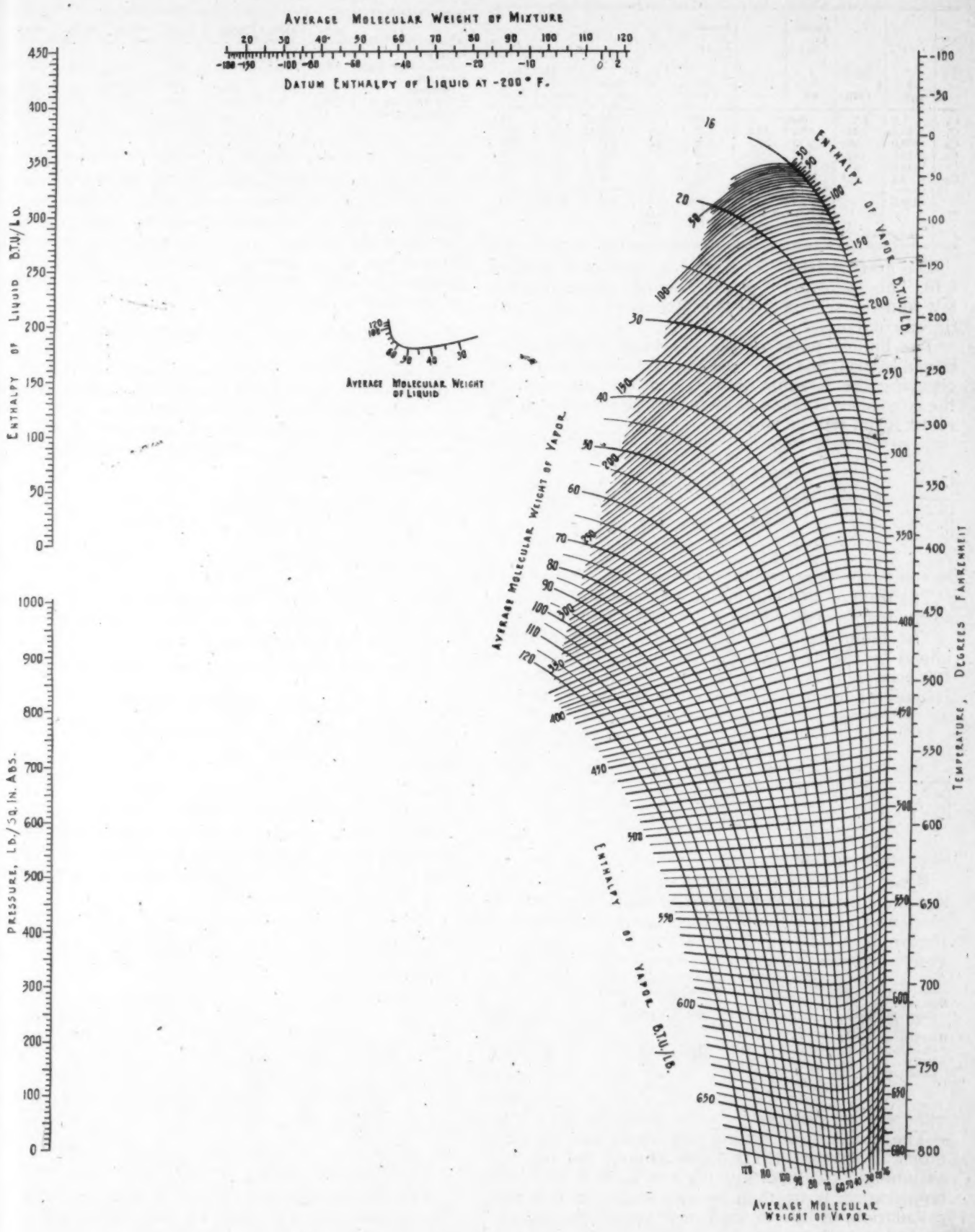
Average Molecular Weight of Mixture
Datum Enthalpy of Liquid at -200° F.
Enthalpy of Liquid B.T.U./Lb.
Average Molecular Weight of Liquid
Pressure, Lb./Sq. In. Abs.
Average Molecular Weight of Vapor
Enthalpy of Vapor, B.T.U./Lb.
Temperature, Degrees Fahrenheit
Average Molecular Weight of Vapor

FIGURE 3

Enthalpy Nomograph for Light Hydrocarbons.

The total liquid is obtained by adding up the total vapor and subtracting from the total feed.

	f m/hr.	K 220°F. 100lb	Assume $\frac{L}{V}=2$ L/VK	v	Assume $\frac{L}{V}=3$ L/VK	v	Vapor lb./hr.	LIQUID m/hr.	LIQUID lb./hr.
C_3..	1.0	4.5	.445	.7	.666	.6	26	.4	18
C_4..	40.0	1.96	1.02	19.8	1.53	15.8	916	24.2	1404
C_5..	23.0	.93	2.15	7.3	3.23	5.4	388	17.6	1268
C_6..	16.0	.45	4.45	2.9	6.66	2.1	180	13.9	1196
C_7..	12.0	.21	9.53	1.1	14.3	.8	80	11.2	1120
C_8..	8.0	.105	19.05	.4	28.6	.3	34	7.7	876
	100.0		V=	32.2	V=	25.0	1624	75.0	5882
			L=	67.8	L=	75.0			
			L/V=	2.10	L/V=	3.00			

The first trial L/V of 2 gave a calculated value of 2.10. This indicated that the assumed value was too low and the second trial of 3.0 checked exactly with the calculated value.

The bubble point of the overhead is also determined by trial and error, assuming a temperature and checking the K-value of Σm. The pressure drop in the condenser is taken as 10 psi so the pressure in the reflux drum will be 90 psia.

	Overhead m/hr.	K 135° F. 90 lb.	mK
C_3	1.0	2.6	2.6
C_4	39.4	.96	37.8
C_5	.4	.36	.1
	40.8		40.5

The total of 40.5 checks the 40.8 moles of overhead so the temperature of the reflux drum will be 135° F.

The temperature of the top tray vapor is determined by the dew point of the overhead product at 100 psia. This is also determined by trial and error, assuming a temperature and checking the Σv/K value.

	Overhead m/hr.	K 145° F. 100 lb.	v/K	PROPERTIES OF TOP TRAY LIQUID Lbs.	M. W.
C_3	1.0	2.6	.4	18	....
C_4	39.4	1.00	39.4	2285	....
C_5	.4	.38	1.0	72	....
	40.8		40.8		58.3

Based on these temperatures, it is possible to calculate the condenser duty from the heat balance on the condenser. The enthalpy values are determined from Figure 3.

	m/hr.	M. W.	lb./hr.	Temp.	Press.	Btu/lb.	Btu/hr.
Heat In:							
Top-Tray Vapor	164.2	57.9	9432	146	100	268	2,530,000
Heat Out:							
Overhead	40.8	57.9	2358	135	L	124	293,000
Reflux	122.4	57.9	7074	135	L	124	878,000
Condenser Duty							1,359,000
							2,530,000

The quantity of second-tray vapor can be determined from the heat balance around the top tray, assuming a temperature for the second tray. This temperature must then be checked by a dew-point calculation of the second-tray vapor. Assuming a second-tray temperature of 150° F., the following heat balance is obtained around the top tray and the condenser. A preliminary calculation indicates that the molecular weight of the second-tray vapor will be 58.2, and this is used to determine the enthalpy from Figure 3. Inspection of this figure indicates that a small error in estimating this molecular weight will not have any noticeable effect on the enthalpy.

	m/hr.	M. W.	lb./hr.	Temp.	Press.	Btu/lb.	Btu/hr.
Heat In:							
Second-Tray Vapor	164.0	58.2	9548	150	100	271	2,586,000
Heat Out:							
Overhead	40.8	57.9	2358	135	L	124	293,000
Top-Tray Liquid	123.2	58.3	7190	146	L	130	934,000
Condenser Duty							1,359,000
							2,586,000

In the above heat balance when all the enthalpy values have been determined from Figure 3, the quantities can be calculated easily by observing that the overhead product enters the system as part of a stream having an average enthalpy of 271 Btu per pound. Thus, the part of the condenser duty applied to condensing the overhead is

$$2358 \times (271 - 124) = 347{,}000 \text{ Btu/hr.}$$

The balance of the condenser duty supplied the reflux from the top tray and this stream also entered as part of a stream having an average enthalpy of 271 Btu per pound and leaves with an enthalpy of 130 Btu per pound. Thus, the quantity of top tray liquid can be calculated

$$\frac{1{,}359{,}000 - 347{,}000}{271 - 130} = 7{,}190 \text{ \#/hr.}$$

This quantity can be used to check the heat balance and the molecular quantities and molecular weights can be calculated by the material balance on the system. The assumed second-tray-vapor temperature is checked by the dew calculation.

	Overhead m/hr.	Top-Tray Liquid m/hr.	Second-Tray Vapor m/hr.	K 150° 100 lb.	v/K
C_3	1.0	1.2	2.2	2.7	.8
C_4	39.4	119.0	158.4	1.04	152.3
C_5	.4	3.0	3.4	.41	8.3
	40.8	123.2	164.0		161.4

The exact dew point is 148° F. which is within 2° of the assumed temperature. In practice, an agreement within 5° can be considered as a satisfactory check in design calculations.

The bubble point of the bottoms at 100 psia gives the temperature at which this stream leaves the column. This is determined by trial and error as previously described.

	Bottoms m/hr.	K 290° F. 100 lb.	m. K.	K 278° F. 100 lb.	m. K.	PROPERTIES OF REBOILER VAPOR Lbs.	M. W.
C_4	.6	.3.3	2.0	3.1	1.9	110	....
C_5	22.6	1.75	39.6	1.60	36.2	2610	....
C_6	16.0	.95	15.2	.86	13.8	1188	....
C_7	12.0	.51	6.1	.45	5.4	540	....
C_8	8.0	.28	2.2	.25	2.0	228	....
	59.2		65.1		59.3	4676	78.9

The first assumption of 290° F. was too high and the second trial was made by calculating the necescary K-value for the n-pentane which is the most important component in the reboiler vapor, which would check the ΣmK value. The desired K-value was 1.59 and the temperature of 278° F. at which the K-value of n-pentane is 1.60 was chosen for the

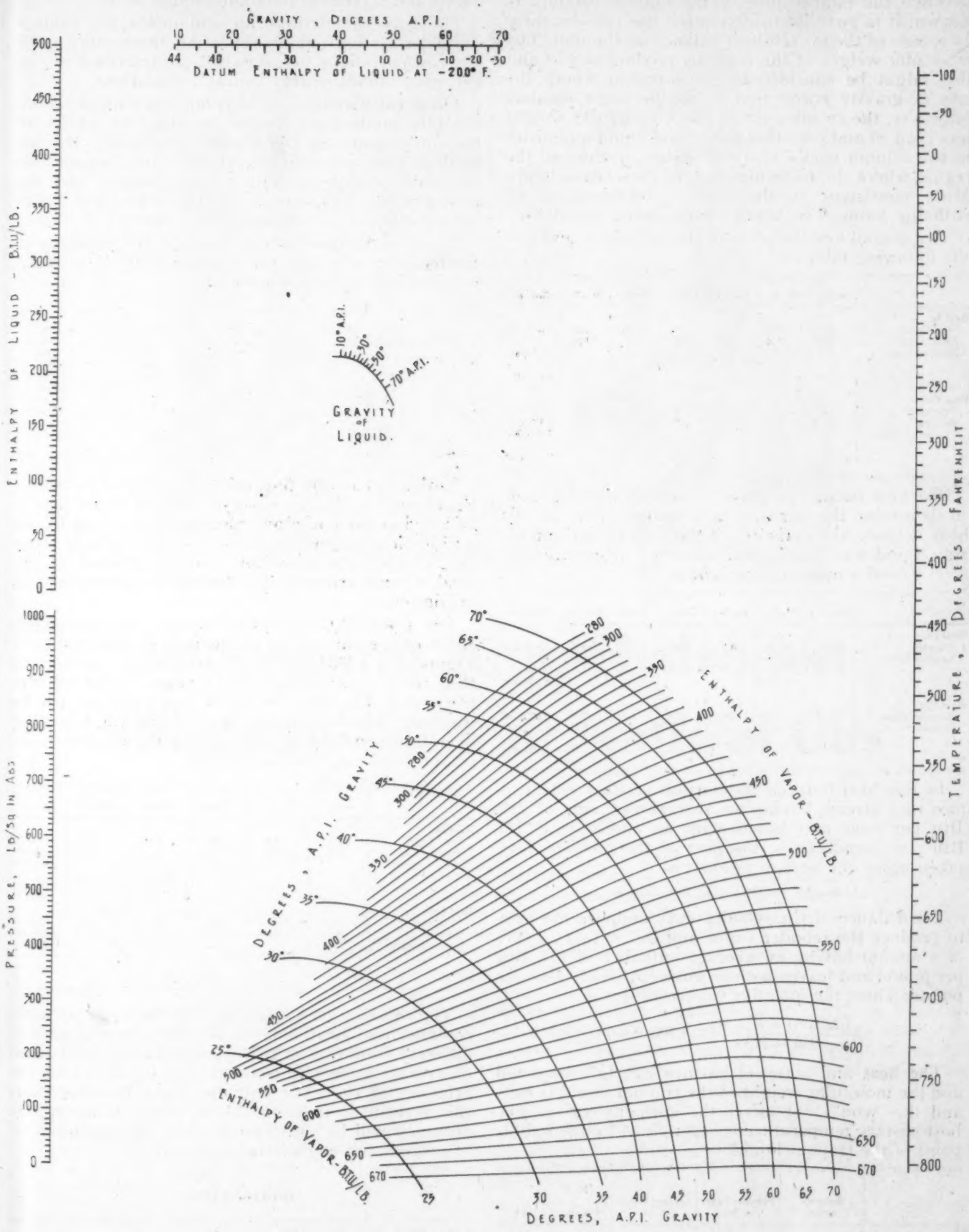

FIGURE 4
Enthalpy Nomograph for Petroleum Fractions. (Based on Characterization Factor 11.8.)

second trial. This can be seen to give a very good check.

When the temperature of the bottom product is known, it is possible to determine the reboiler duty by means of the overall heat balance on the unit. The molecular weight of the bottoms product is 86.9 and this might be considered in the region where the specific-gravity correlation would be more reliable. However, the reboiler vapor has a molecular weight less than 80 and the other vapor and liquid quantities in the column would also fall within or close to the region where the molecular weight correlation holds. More consistent results will be obtained if all enthalpy values are based on the same correlation.

The overall heat balance on the column is given in the following table:

	m/hr.	M. W.	lb./hr.	Temp.	Press.	Btu/lb.	Btu./hr.
Heat In:							
Feed Vapor	25.0	65.0	1624	220	100	308	501,000
Feed Liquid	75.0	78.4	5882	220	L	189	1,111,000
Reboiler Duty							1,229,000
							2,841,000
Heat Out:							
Overhead	40.8	57.9	2358	135	L	124	293,000
Bottoms	59.2	86.9	5148	278	L	231	1,189,000
Condenser Duty							1,359,000
							2,841,000

The heat balance on the reboiler can then be made to determine the quantity of reboiler vapor. In this heat balance, the enthalpy of the trapout or bottom-tray liquid was based on an assumed temperature of 255° F. and a molecular weight of 82.

	m/hr.	M. W.	lb./hr.	Temp.	Press.	Btu./lb.	Btu/hr.
Heat In:							
Trapout	16.0	81.8	13648	255	L	215	2,935,000
Reboiler Duty							1,229,000
							4,164,000
Heat Out:							
Reboiler Vapor	107.8	78.9	8500	278	100	350	2,975,000
Bottoms	59.2	86.9	5148	278	L	231	1,189,000
							4,164,000

In this heat balance the bottom product enters as part of a stream having an average enthalpy of 215 Btu per hour and leaves with an enthalpy of 231 Btu per pound. Thus, the part of the reboiler duty taken up by the bottom product is:

$$5148\ (231 - 215) = 82{,}000\ \text{Btu/hr.}$$

The balance of the reboiler duty supplies the heat to produce the reboiler vapor and this enters as part of a stream having an average enthalpy of 215 Btu per pound and leaves with an enthalpy of 350 Btu per pound. Thus, the quantity vaporized is:

$$\frac{1{,}229{,}000 - 82{,}000}{350 - 215} = 8{,}500\ \text{lbs./hr.}$$

The heat and material balance can be completed and the molecular weight of the trapout liquid is 81.8, and this would not affect the enthalpy value. The bottom-tray temperature is determined by the bubble point of the trapout liquid.

	Bottoms m/hr.	Reboiler Vapor m/hr.	Trapout Liquid m/hr.	255° F. K 100 lb.	mK
C_4	.6	3.4	4.0	2.6	10.4
C_5	22.6	65.8	88.4	1.30	114.9
C_6	16.0	25.1	41.1	.67	27.5
C_7	12.0	9.9	21.9	.34	7.4
C_8	8.0	3.6	11.6	.18	2.1
	59.2	107.8	167.0		162.3

The exact bubble point of the trapout is 257° F. at which the K-value of C_5 is 1.33, and this is well within the maximum tolerable limit of 5° F.

The tray calculations up and down the column could be carried out by repeating these steps until the concentrations are matched as described in the various methods of tray-to-tray calculations.

These calculations can be compared with the more accurate method previously described in which all the components are considered individually. By this method, the condenser duty is the same because the overhead product is almost pure butane and the nomographs of Figures 2 and 3 give identical latent heats for butane at its molecular weight of 58.

As an illustration of this method, the enthalpy of the feed vapor at zero pressure and 220° F. is calculated as follows from Figure 2.

	lb./hr.	Btu/lb.	Btu/hr.
C_3	26	370	9,600
C_4	916	359	329,000
C_5	388	356	138,200
C_6	180	354	63,600
C_7	80	354	28,300
C_8	34	354	12,000
	1624	357	580,700

In this calculation the enthalpy values of C_7 and C_8 lie off their scales, however, it will be noted that in this region the enthalpy values of C_6, C_7 and C_8 are all identical at zero pressure, and since these latter two components constitute only 7 percent of the total, a small error in their enthalpy values would be negligible.

For a gas of 65 molecular weight, the isothermal effect of pressure up to 100 pounds determined from Figure 3 is $(308 - 318) = -10$ Btu per pound and thus the total enthalpy of the vapor is 564,500 Btu per pound. The enthalpy of the liquid streams can be similarly calculated from Figure 2 and the following overall heat balance is obtained on the column based on the previous condenser duty.

	m/hr.	lb./hr.	Temp.	Press.	Btu/lb.	Btu/hr.
Heat In:						
Feed Vapor	25.0	1624	220	100	347	564,500
Feed Liquid	75.0	5882	220	L	221	1,300,500
Reboiler Duty						1,215,400
						3,080,400
Heat Out:						
Overhead	40.8	2358	135	L	175	413,000
Bottoms	59.2	5148	278	L	254	1,308,400
Condenser Duty						1,359,000
						3,080,400

The reboiler duty calculated by the more accurate componential heat balance is only about 1 percent different from that of the approximate method based on average molecular weights. This is well within the accuracy of the basic enthalpy data. In other cases the agreement may not be so close, however, the accuracy will be sufficient to meet the requirements of process and engineering calculations.

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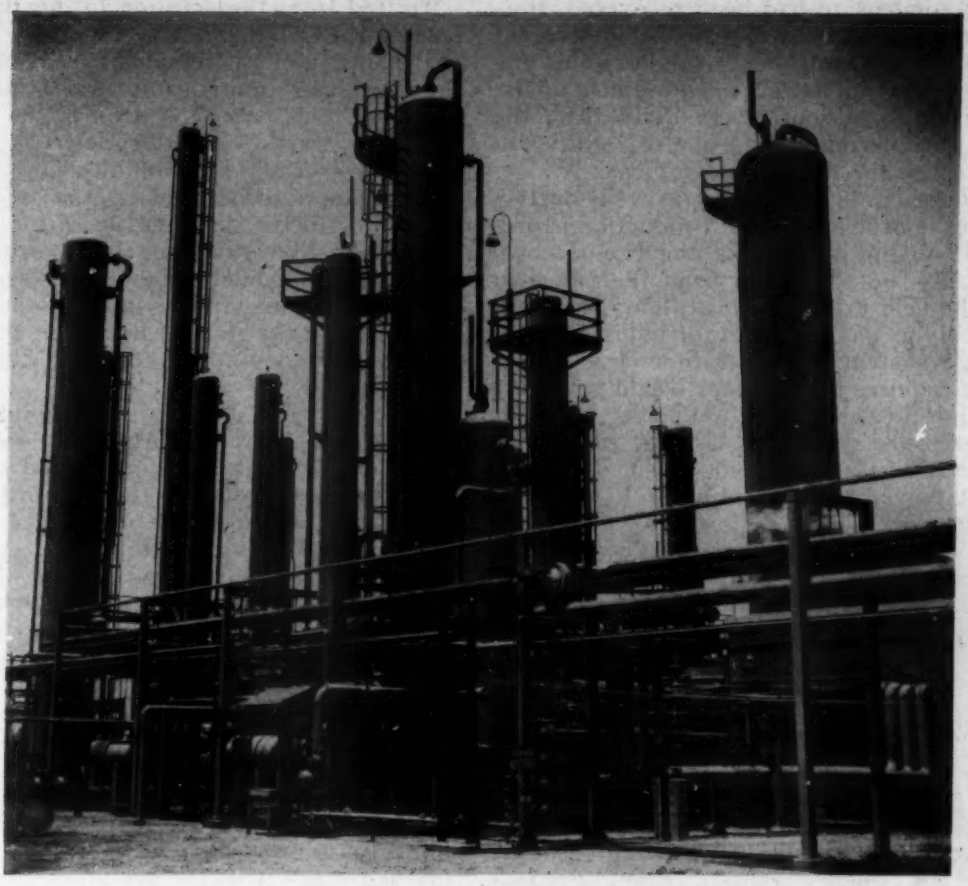


Processing High-Hydrogen Sulphide-Content Natural Gas

C. C. PRYOR, Associate Editor

IN THE operation of its natural gasoline plant near Stamps, Arkansas, McKamie Gas Cleaning Company seeks high yield of residue gas, since its primary market is to industrial and domestic gas consumers. Original design of the plant included presaturation of its absorption unit, so as to leave the maximum of fuel gases in the plant residue.

In the McKamie field three-stage separation is required to separate the sour gases from distillate. Consequently recompression of the total field stream is essential prior to processing. But the presaturation process leaves it unnecessary to recompress the plant residue for delivery, after flashing, into the pipe line.

McKamie Gas Cleaning Company, owned jointly by Standard Oil Company (New Jersey) and The Atlantic Refining Company, was formed to operate a plant with capacity of 30,000,000 cubic feet of gas per day, and to recover 32,000 gallons of liquids daily. In addition to its presaturation absorption unit, the plant includes a desulphurization unit and a dehydration unit as well as the required fractionation units.

High hydrogen sulphide content of the gas resulted in the construction of a sulphur conversion unit nearby by Southern Acid & Sulphur Company. Recovery from the desulphurization unit goes to this plant where pure sulphur is processed.

Essentially the presaturation process consists of contacting lean absorption oil with methane, ethane and propane in a 36-inch 16-tray presaturator tower. This presaturated absorption oil is pumped over the main high-pressure tower to contact incoming gas. Since the oil is presaturated it does not pick up methane, ethane and propane from the main gas stream. These remain in the high-pressure residue gas stream, which is dehydrated before entering the pipe line.

The process eliminates large volumes of low-pressure residue gas and the compressors that would be necessary to recompress the gas back to the absorber residue gas pressure. The oil rate is less than that necessary for absorption without presaturation, because of the reduced temperature of absorption in the main absorber.

Natural gas in the McKamie field has an unusually high hydrogen sulphide content, 4300 grains per 100 standard cubic feet, and a high condensate ratio. In the early days of the field, residue gas was burned in

flares after the condensate had been recovered, thus wasting thousands of barrels of valuable petroleum by-products and large quantities of sulphur. This gas could not be used for industrial sales because of the high hydrogen sulphide content. In early 1943, the field was shut in to permit installation of facilities to conserve and utilize the flared residue gas.

At present there are 20 producing gas wells on 160-acre spacing in the McKamie field. Production is prorated according to the capacity of the H_2S-removal plant; thus, gas is not produced from the reservoir and flared in the field to produce distillate. The wells are produced only as gas is needed for industrial sale. The three-stage system for separation of gas from the condensate requires three separate field gathering systems: a high-pressure system of 650 psig; an intermediate-pressure system of 45 psig, and a low-pressure system with an elevated 2 psig separator.

In the plant flow diagram of Figure 1, the three sour gas streams are labeled A, B, and C. Streams B and C are compressed to 650 psig and flow into the high-pressure stream A to the desulphurization unit at 635 psig. Composition of this combined stream at G is given in the table below:

Gas to Desulphurization at G, 26,500,000 scfd, 0.792 sp. gr.

Components	N_2	CO_2	H_2S	C_1	C_2	C_3	C_4i	C_4	C_5i	C_5	C_6	C_{7+}
Mol Percent	11.64	4.48	7.84	63.87	7.05	2.97	0.72	0.85	0.27	0.14	0.11	0.05

Desulphurization

The gas flows up through the purification absorber, which is packed with Raschig rings, countercurrent to a 15-20 percent aqueous solution of monoethanolamine (MEA) pumped continuously into the tower near the top at about 100° F. at a rate of approximately 420 gallons per minute. Hydrogen sulphide and carbon dioxide are absorbed by the MEA solution, and the purified gas flows at a rate of 23,300,000 cubic feet daily and 0.72 specific gravity with less than 0.05 grains of sulphur per 100 cubic feet from the desulphurization absorber to the gasoline-plant absorber for processing. The rich spent MEA solution flows from the base of each of the two purification absorbers through liquid-level controlled valves, through the rich-lean MEA heat exchangers to the reactivator tower entering near the top at about 255° F. After flowing down through the reactivator tower, the solution is boiled in the reboilers at approximately 285° F. to expel nearly all the hydrogen sulphide and carbon dioxide. The reboiler is heated by exhaust steam from the pump turbines.

Steam generated by boiling the solution in the reboiler passes up through the reactivator, heating the down-coming solution to its boiling point (approximately 285° F. at 35 psig pressure) and carries the hydrogen sulphide, carbon dioxide and steam flow to a condenser, where the steam is condensed and the hydrogen sulphide and carbon dioxide are cooled to about 100° F.

The acid gas, from 3 to 3.5 million cubic feet daily, flows to Southern Acid & Sulphur Company's plant where the pure sulphur is recovered at a rate of 60-65 tons per day. Condensed MEA solution from the condenser flows to a reflux accumulator drum and then is pumped back to the reactivator tower as reflux.

The desulphurization process is a simple continuous-cycle process for separation of acid gas (i.e., hydrogen sulphide and carbon dioxide) from gases and liquids. By scrubbing the gases and liquids with solutions of amines (organic alkalis) the acid gases are removed. Then the absorbed acidic gases are separated from the solution by heating, thus regenerating the solution for further acid gas absorption. If the supply of gas remains approximately the same, the plant operation is completely automatic.

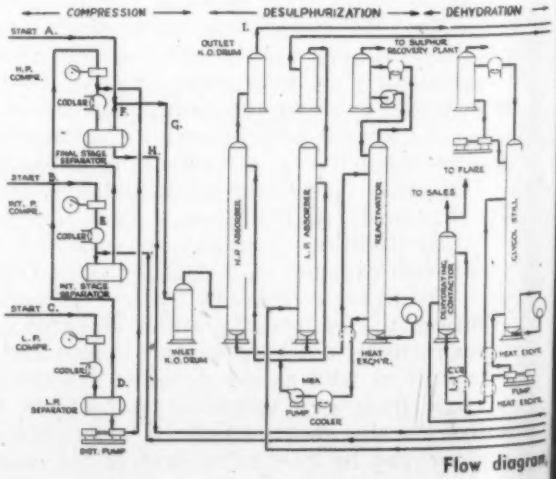
Absorption

Gas overhead (I) from the high-pressure desulphurization absorber flows to the base of the 24-tray, 60-inch diameter absorber, operating at 615 psig and 110° F. Residue gas from the absorber is cooled and passed to the residue scrubber. Gas from the scrubber flows to the dehydrating unit of the plant, and the liquids enter the rich oil line from the bottom of the absorber and flow to the cold vent tank for venting at 385 psig and 108° F.

Hot lean oil, 252 gallons per minute, 35° API, 220 mol percent, from the surge tank is pumped through lean-rich oil heat exchangers and a cooler to the presaturator tower and the reabsorber. Lean oil in the presaturator tower contacts gases from the cold vent tank (M) and the depropanizer reflux accumulator tank (W). The gases from the depropanizer reflux tank are first passed through the low-pressure desulphurization absorber. Then the presaturated absorption oil, having a specific gravity of 0.816, from the tower is cooled and pumped at a rate of 274 gallons per minute and 95° F. to the M&M absorber. Overhead gas from the presaturator flows to the presaturator residue scrubber. Gas from the scrubber is used for fuel, and the liquids enter the feed line to the 42-inch diameter, 20-tray, high-pressure still.

Analysis in mol percent in the table below, of the gas charged to the absorber, residue gas, and the rich-oil stream, illustrates the operation of the presaturation process in the McKamie plant.

CONSTITUENT	Gas Feed Mol Percent (I)	Rich Oil Mol Percent (J)	Residue Gas Mol Percent (K)
N_2	13.27	.....	13.29
C_1	72.84	14.01	73.29
C_2	8.05	6.48	8.72
C_3	3.39	5.83	4.40
C_4i	0.82	2.36	0.22
C_4	0.97	3.20	0.08
C_5i	0.31	1.07	.....
C_5	0.16	0.56	.....
C_6	0.13	0.43	.....
C_{7+}	0.06	0.22	.....
Oil	.....	65.84	.....

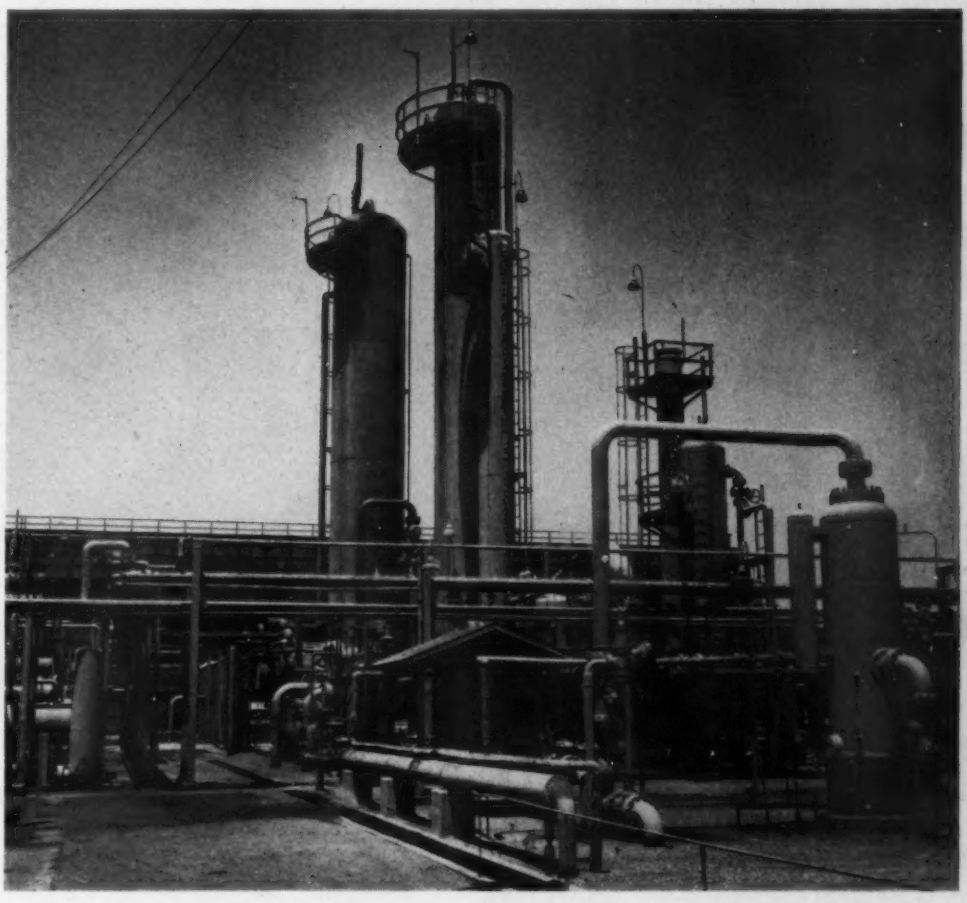


Flow diagram

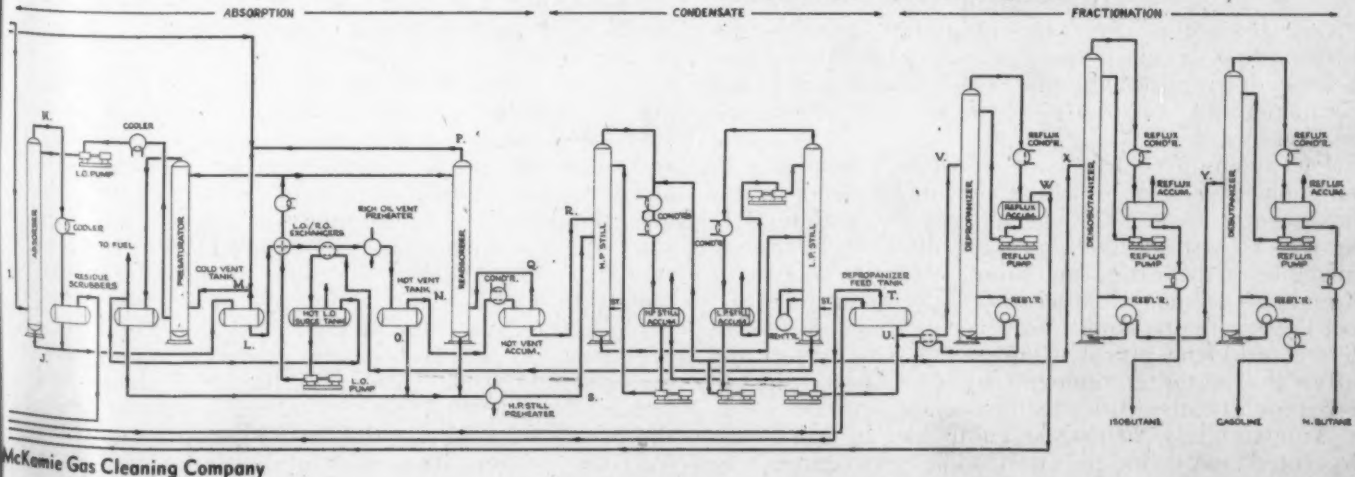
After the rich oil stream (L) from the absorber is vented in the cold vent tank, it flows through the lean-rich oil heat exchangers and a preheater, increasing the temperature to 380° F., to the hot vent tank operated at 185 psig. Vapor (N) from the hot vent tank flows through a condenser to the hot vent accumulator at 180 psig and 95° F. Gases (Q), 1,340,000 cubic feet daily, 1.022 specific gravity, from the hot vent accumulator tank are charged to the reabsorber, and the liquid, 4.4 gallons per minute, to the high-pressure still. Reabsorber residue gas (P), 1,031,000 cubic feet daily, 0.803 specific gravity, is used in the presaturator tower, and the bottoms, 33.4 gallons per minute, 0.787 specific gravity, flow into the high-pressure still charge line. Condensed vapor (O) flows to the high-pressure still through a preheater (S).

Fractionation

Steam stripped bottoms from the high-pressure still, 150 psig and 395° F., are charged to the low-pressure still at 30 gallons per minute and 0.625 specific gravity. Overhead product of the still at 185° F. is condensed and flows to the accumulator for reflux at 25 gallons per minute, 0.585 specific gravity, to the tower, and for charging at 63.7 gallons per minute, 0.648 specific gravity, to the depropanizer feed tank (U) and the depropanizer tower (V). Vapor from the depropanizer feed tank is returned (T) to the final stage of compression with the incoming gas to the plant at 400 psig, and condensate from the final stage of compression flows to the feed tank. Overhead vapor from the low-pressure still, which is the same size as the high-pressure still, is condensed and collected in the accumulator drum. The low-pressure still is operated at 20 psig, top temperature of 200° F. and bottom temperature 390° F. A portion of the liquid from the accumulator is pumped over to the reflux condensers of the high-pressure still, and the remainder is refluxed to the low-pressure still. Lean oil from the low-pressure still is returned to the hot lean oil surge tank at 279 gallons per minute and 0.85 specific gravity. Vapor (W) from the depropanizer reflux accumulator flows to the low-pressure desulphurization absorber for purification and subsequent charging to the presaturator; the liquid, 45 gallons per minute, 0.527 specific gravity, is refluxed to the depropanizer tower. The 42-inch diameter depropanizer tower has 10 trays spaced 18 inches and 20 trays, spaced 24 inches apart, and is operated at 350 psig pressure with 128° F. top temperature and 283° F. bottom temperature. Feed (X), 24.4 gallons per minute, 0.607 specific gravity, to the deisobutanizer tower is taken from the depropanizer reboiler, which is divided so that a portion, 44 gallons per minute,

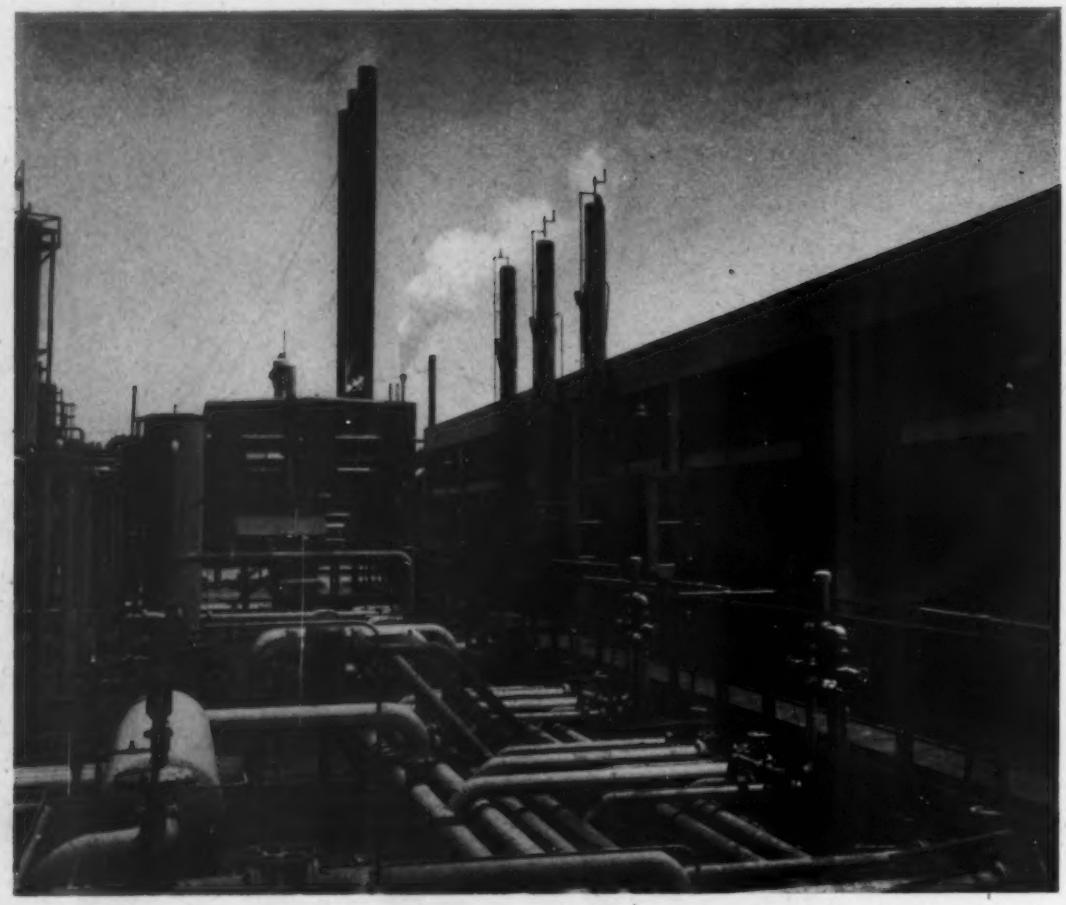


Desulphurization and dehydration units. The incoming gas separator is at right, and meter house in center foreground.



McKamie Gas Cleaning Company

flows into the line to the high-pressure still condensers. Isobutane is produced overhead from the deisobutanizer, and the bottoms (Y) charged to the debutanizer at 18 gallons per minute, 0.621 specific gravity. The deisobutanizer is a 42-inch-diameter tower with 65 trays. It operates at 85 psig, 128° F. top and 196° bottom temperature. Reflux of the deisobutanizer is 58 gallons per minute, 0.565 specific gravity, and the debutanizer, 20 gallons per minute of 0.584 specific gravity. The plant is designed to produce 8261 gallons per day of isobutane of 0.565 specific gravity, when operating at capacity; 13,998 gallons per day of 0.647 specific gravity gasoline, and 9684 gallons per day of 0.584 specific gravity butane.



Compressor house and mainfold piping for the three stages of compression for incoming gas. Boiler building is in background.

Dehydration

Gas from the plant for industrial sales is dehydrated in a diethylene glycol dehydration unit before entering the pipe line. The unit consists of the contactor and still towers, scrubber, heat exchanger and pumps.

Diethylene glycol is made up of two molecules of ethylene glycol from which one molecule of water has been removed. The specific gravity is about 1.12 at 68° F. with a boiling point of 473° F. Freely miscible with water, methanol, ethanol, and other liquids at ordinary temperatures, the diethylene glycol is very hydroscopic; however, it does not hydrolize to ethylene glycol when mixed with water, and after the water is removed by distillation, the diethylene glycol returns to its normal state.

Appoximately 20,000,000 cubic feet of gas is dehydrated daily in the unit. The gas enters the dehydrating contactor at 95° F. and is contacted by 12 gallons per minute of glycol entering the top at 100° F. The contactor is 48 inches in diameter with 6 trays spaced 24 inches apart. Spent solution leaves the bottom of the contactor at 105° F. and flows thrpugh two heat exchangers, where it cools the fresh glycol going to the contactor from 280° F. and increases its own temperature to 245° F. before entering the 24-inch diameter glycol still. Heat to the still is supplied by a reboiler which operates the still at 280° F. bottom temperature and 245° F. top temperature. Water vapor is taken overhead and condensed and 2 gallons per minute is refluxed to maintain the correct temperature at the top of the towers. Reclaimed glycol is drawn from the bottom of the still for re-use. The dewpoint of the gas dehydrated is about 30° F., which is well within the limits required by the pipe line.

The success of the companies involved in solving the problems of converting sour gas into such products as isobutane, normal butane, gasoline, sulphur, and sweet dry gas is a distinct conservation measure and an economic utilization of gas resources. In addition, the industrial development of the area has been sped by an adequate supply of fuel. During the first two years of operation, the plant processed approximately 14 billion of the cumulative production of the field of 30 billion cubic feet (to pass the lead acetate test) for sale as merchantable gas. The plant manufactured 20,700,000 gallons of liquid products, and produced 2,046,000 cubic feet of acid gas from which the Southern Acid & Sulphur Company made 32,126 long tons of pure elemental sulphur.

Regeneration of Spent Catalyst In Fluid Catalytic Cracking

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THE factors which influence the design, operation, and control of spent-catalyst regeneration facilities in commercial fluid catalytic cracking are discussed. The regeneration of spent cracking catlyst is accomplished by burning about 1 to 2 weight-percent of carbonaceous material from the catalyst under closely-controlled operating conditions. During the combustion a large quantity of heat is produced. Regeneration supplies a portion of the heat required to support the reaction in the catalytic conversion zone, and excess heat is used to produce steam. A description of the facilities used for regeneration, and the integration of these facilities with the rest of the catalytic plant, is presented.

The paper was contributed by the Fuels Division of The American Society of Mechanical Engineers, and presented at the Spring Meeting of the society, at Tulsa, March 2-5, 1947.

THE regeneration of spent catalyst by removal of carbonaceous deposit under closely-controlled combustion conditions is an important feature of the fluid catalyst technique which has gained wide acceptance during the past six years. Regeneration is important not only because of the necessity for restoring catalyst activity, but also because of the large heat effects involved. In spite of the fact that only about 1 to 2 weight-percent of carbonaceous material is present on the catalyst, the combustion in the regenerator contributes a major portion of the heat required to support the conversion reaction taking place in the reaction section of the catalytic plant, and residual heat usually is used to make steam. The fluid catalyst type of operation currently is being used, or has been proposed for application, in many chemical and petroleum catalytic conversion processes where continuous, rather than intermittent, operation is desired. The most extensive application to date has been in the petroleum industry, where fluid catalytic cracking is a well established process. Fluid catalytic cracking will be used as a basis in the following for discussion of regeneration design and practice.

Description of Process

Descriptions of the oil, air, and catalyst flows in the several types of fluid catalytic cracking systems have been presented in many papers published during the past few years. The general description which follows is a brief review for the purposes of the present paper.

The process equipment comprising a fluid catalyst unit can be divided into four principal sections: feed-preheat section, reaction section, regeneration section, and product-fractionation section. Figure 1 is a schematic drawing showing these sections and may be referred to in tracing the process flow.

In the feed-preheat section fresh charging stock (gas, oil, etc.) is heated in tubular heat exchangers with hot streams from the fractionator and, in some cases, by means of an oil- or gas-fired furnace. Feed, either partially or completely vaporized, is directed to the regenerated-catalyst pickup point where it contacts hot, finely divided, solid catalyst particles. This mixture of oil and catayst flows upward into the reaction section where the cracking reaction takes place. A fluidized bed of catalyst is maintained in the reactor. Cracked vapors pass overhead from the reactor catalyst bed through cyclone separators, where all but a trace of catalyst is removed, and then these vapors enter the fractionation section. In this section products are separated according to desired boiling ranges, and the small residual amount of catalyst is recovered for return to the catalytic system.

During the cracking reaction a catalyst deposit, or coke, is formed on the catalyst. This material must be removed in order to maintain catalyst activity and selectivity. To accomplish this regeneration, spent catalyst is continuously drawn from the reactor and is directed through a steam stripping zone where entrained and volatile hydrocarbons are removed, and is then caused to flow through a standpipe. At the bottom of the spent catalyst standpipe the catalyst meets a stream of air which carries it into the regeneration section. A fluidized bed of catalyst is established within the regenerator, as in the reactor. The temperature of the burning which occurs within the regenerator is controlled by continuously drawing a stream of catalyst from the regenerator, cooling this material in a vertical tube exchanger, and returning cooled catalyst to the regenerator. Steam is produced in this recycle catalyst cooler; or, the heat may be used for heating process oil streams.

As the regeneration progresses, sufficient coke is burned from the catalyst to restore its activity, and the regenerated material is returned continuously to the cracking zone by means of another standpipe and the oil-transfer system previously described. The gaseous combustion products of regeneration pass upward through the regenerator through catalyst-recovery equipment, and then are vented to the atmosphere. In many instances the flue gas leaving the regenerator passes through tubular boilers for additional heat recovery in the form of steam.

Regeneration Variables

The principal variables affecting the design of a fluid catalyst regeneration system are: Temperature of regeneration, type of catalyst, analysis of catalyst deposit, fraction of catalyst deposit removed during

regeneration, contact of air and catalyst and catalyst residence time, and regeneration pressure. These factors are discussed in general below.

Temperature of Regeneration

Commercial fluid catalytic cracking units operate with regeneration temperatures ranging from slightly below 1000° F. to about 1200° F. The choice of operating temperature for design purposes is a balance between several factors. In general, the higher the temperature, the more readily the combustion occurs. On the other hand, at the highest temperature levels, permanent catalyst deactivation occurs at an accelerated rate, and construction problems and costs tend to become more important considerations. The type of catalyst to be employed also is to be considered, since some are more heat-resistant as regards activity decline than others. Much study has been given these factors, and their combined effects have been valuated, with the result that current designs generally call for a regeneration temperature falling within the 1025-1100° F. range.

Type of Catalyst

As mentioned in the foregoing, the type of catalyst to be used affects the design and operation of regeneration equipment because of temperature deactivation problems. The catalyst type also will affect regeneration-air requirements and heat evolution, since catalysts having different chemical compositions result in various ratios of CO_2 to CO in effluent regeneration gases. With various commercial catalysts the CO_2-to-CO ratio varies between about 50:50 to 65:35. The presence of certain metallic contaminants, such as iron, which may accumulate on the catalyst during operation, tends to increase the CO_2-to-CO ratio, and results in increased air requirements for burning of any given amount of catalyst deposit.

Analysis of Catalyst Deposit

The coke, or catalyst deposit, which is to be removed from the catalyst during regeneration is composed principally of carbon and hydrogen, but also may include sulfur. The actual composition of the deposit is complex as regards hydrocarbon types and is not well known. Depending upon the adequacy of the spent-catalyst steam-stripping facilities, which are intended to remove entrained and volatile hydrocarbons, leaving only coke, the carbon content of sulfur-free deposit varies from about 93 to 87 weight-percent, and hydrogen content from about 7 to 13 weight-percent. (Hydrogen content calculated by nitrogen balance across the regenerator and determined by oxygen disappearance. Since about four times as much air is required to burn one pound of hydrogen as is necessary for one pound of carbon, the importance of adequate spent-catalyst stripping facilities and low hydrogen content in determining regeneration-air requirements is obvious. Normally, with a well-designed spent-catalyst stripper, slightly less than 10 weight-percent of hydrogen in coke can be specified for design purposes. Heat of combustion and

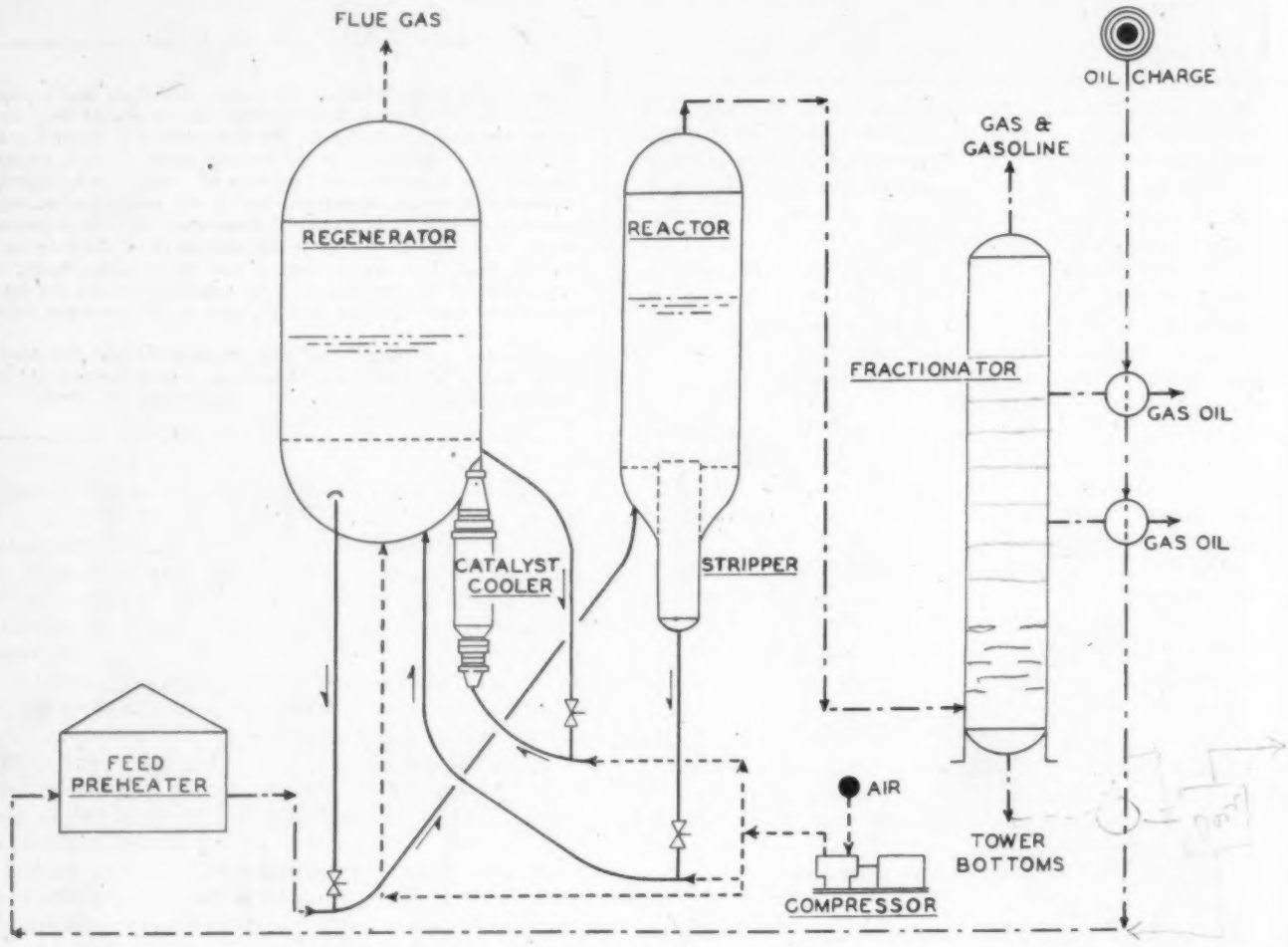


FIGURE 1

regeneration-air requirements for various CO_2-to-CO ratios and hydrogen contents are covered in a subsequent section.

Fraction of Catalyst Deposit Removed During Regeneration

The weight-percent of catalyst deposit on spent catalyst charged to the regenerator varies between less than 1 to more than 2 percent in commercial installations, and is partially a function of the catalyst-to-oil ratio fed to the cracking zone. Regenerated catalyst contains from about 0.3 to 1.0 weight-percent of residual deposit after completion of combustion. A balance between several factors defines the coke level desired on regenerated and spent catalyst for any specific design. First, the burning of one pound of coke from catalyst having a high deposit content, generally proceeds more readily than the burning of the same amount of coke from catalyst having a low coke content. Second, it is desirable from the standpoint of the cracking-reaction side of the process to maintain a relatively low catalyst carbon content. Third, high coke percentages on catalyst in the regenerator are not satisfactory, since catalyst entrainment in flue gas tends to become an increasingly important factor. In commercial operation, balancing between these factors has established the desirability of maintaining coke content of regenerated catalyst at between about 0.3 and 0.7 weight-percent by control of other operating variables.

Contact of Air and Catalyst and Catalyst Residence Time

One of the more important factors affecting the economical utilization of regeneration air is the provision of a suitable distributing device to promote adequate contacting of air and catalyst within the regenerator. Distribution of air, typically, is accomplished by means of a grid plate located at the bottom of the regenerator-catalyst bed. This grid contains many small holes and imposes 0.5 to 1.0 pounds pressure drop to the air flow. It is also desirable to avoid a shallow catalyst bed within the regenerator through which mass channeling of air may occur. In large commercial units, at least 10, and preferably 15, feet of regenerator-bed depth are desired when the regenerator takes the form of a single bed.

Gas should flow through the regenerator at low velocity. Commercial designs call for about 1.5 feet per second, which is satisfactory from the standpoint of minimizing catalyst entrainment in the gas.

The residence time of catalyst within the regenerator has been varied over exceedingly wide ranges in test work on commercial units. With good distribution of air there appears to be no appreciable incentive for extending the residence time beyond 5 minutes when attempting to control to approximately 0.5 weight-percent carbon on regenerated catalyst. Because of the effect of other necessary design factors (required bed depth for proper air distribution, maximum allowable gas velocity in regenerator, etc.) the residence time ordinarily exceeds this minimum value and ranges between 10 and 20 minutes.

Regeneration Pressure

Commercial fluid-cracking-unit regenerators operate with pressures at the tops of the vessels ranging between about 1 and 13 pounds per square inch, gage. Obviously, with specified maximum allowable gas velocities within the regenerators, the higher pressures permit smaller vessels in new designs, or greater air flows in existing units. It has been shown that the combustion of catalyst deposit proceeds more favorable at the high pressure levels. The choice of pressure used weighs these factors and others against the increased cost of compressing large amounts of air to the higher pressure levels.

Heat Release and Air Requirements

The amount of heat released in the burning of carbonaceous material transferred with catalyst from the reactor to the regenerator is influenced by the several factors which control regeneration operation. For design purposes, the amount of heat released per pound of catalyst-deposit burned can be calculated from the expected flue-gas analysis, hydrogen content of coke, etc., each of which follows the general concepts mentioned above under "Regeneration Variables." Table 1 summarizes net heat of combustion values for several possible conditions.

It is to be noted that the weight-percent of hydrogen in catalyst deposit is designated as that amount determined by oxygen disappearance calculations based on Orsat analyses and nitrogen balance. Oxygen is probably also consumed in the regenerator oxidizing metallic impurities which are in the catalyst. In some cases account is taken of this factor, but in most instances it is neglected due to lack of specific knowledge as to the magnitude of the adjustment to be applied.

Net heat-of-combustion values are used in establishing an over-all heat balance around the regeneration system. The amount of heat which must be removed in the form of steam is determined by difference after determining the amount of heat transferred to the reaction zone due to the rise in catalyst temperature in passing through the regenerator and the net heat leaving the regenerator in combustion gas. As an example of the distribution of combustion heat in a specific commercial unit, which will be discussed in more detail later, the data of Table 2 are presented. In this unit, construction and design details are such that approximately 53 percent of the heat released in burning is recovered in recycle-catalyst coolers as steam. In other types of design as much as 65 or 70 percent of the heat is recovered in producing steam; in some, no heat is recovered in the form of steam, control of the regeneration system being obtained by absorbing in the reactor system all heat recoverable from catalyst.

Combustion air requirements for the regeneration

TABLE 1

Heat of Combustion of Catalyst Deposit in Fluid Catalytic Cracking

Ratio CO_2/CO in Combustion Gas	Weight Percent of Hydrogen in Catalyst Deposit*	Net Heat of Combustion of Deposit Burned from Catalyst-Btu./Lb. of of Deposit
50:50	8	12,440
65:35	8	13,840
50:50	12	14,140
65:35	12	15,480

BASIS

Reaction	Net Heat of Combustion Btu./Lb. of H or C
$H + \frac{1}{4}\ O_2 \longrightarrow \frac{1}{2}\ H_2O$	51,650
$C + O_2 \longrightarrow CO_2$	14,100
$C + \frac{1}{2}\ O_2 \longrightarrow CO$	3,960

* Measured by oxygen disappearance in regenerator as determined from Orsat analysis and nitrogen balance.

TABLE 2

Distribution of Regeneration Heat in a Commercial Fluid Catalytic Cracking Unit

	Millions of Btu./Hr.	Percent
Heat released	238	100
Heat in Flue gas (over entering air)*	56	23
Heat to reaction zone	36	15
Heat to steam	125	53
Heat loss and unaccounted for	21	9
Total	238	100

BASIS

Operation shown in Table 4.
Net heat of combustion of coke is 13,840 Btu./Lb. at 8 percent hydrogen
Reaction zone at 900° F.
Steam produced at 160 psig from 300° F. boiler-feed water.

* Approximately 50 percent of this heat potentially recoverable in a flue-gas boiler, bringing steam to 153 millions of Btu./Hr. or 64 percent of heat release.

operation are affected by all factors which influence dry flue gas composition, by the hydrogen content of catalyst deposit, and by the oxygen content of effluent flue gas required for control. Table 3 summarizes air requirements calculated for various ratios of CO_2/CO in flue gas, various hydrogen contents of the coke, and for 2 percent oxygen in flue gas. Actual practice confirms closely the stoichiometrically calculated requirements.

Control of Regeneration

Many variations in regeneration-control technique have been used in fluid-catalytic-cracking units. The system described in the following is typical of practice used in several instances.

The flow of spent catalyst from the spent-catalyst standpipe, and the consequent load imposed upon regeneration facilities, is regulated by means of a slide valve. The opening of the valve is controlled automatically by positioners which are controlled by the level of catalyst in the reactor. A specified catalyst level is maintained in the reactor so that cracking severity, and coke production, are maintained constant.

The air used to transport catalyst from the bottom of the spent-catalyst standpipe to the regenerator is flow controlled at the quantity which will give suitable flow characteristics and maintain satisfactorily low burning rate in the carrier line. Additional combustion air enters the regeneration zone through the recycle-catalyst-cooler system and through auxiliary air lines. Each of these flows also is controlled at desired rates. The speed of the air compressor supplying combustion air is regulated by a controller which gives constant pressure at the discharge of the machine.

Temperature in the regenerator catalyst bed is maintained at a specified level by circulating hot catalyst through a standpipe, through a cooler, and then back to the regenerator. The rate of catalyst circulation through the cooler is automatically regulated by temperature controllers which actuate slide-valve positioners. Two circulating loops are used on large units. The boiler-feed water and steam systems appurtenant to the recycle-catalyst coolers ordinarily are controlled automatically.

TABLE 3

Combustion Air Requirements in Fluid Catalytic Cracking

Ratio CO_2/CO in Combustion Gas	Weight Percent Hydrogen in Catalyst Deposit*	Combustion Air Requirement Lbs. Air/Lb. Deposit Burned
50:50	8	12.05
65:35	8	12.92
50:50	12	13.18
65:35	12	14.02

BASIS

2 percent in dry effluent flue gas.
Air supplied at 100° F., 50 percent relative humidity

* Measured by oxygen disappearance in regenerator as determined from Orsat analysis and nitrogen balance.

In regeneration operations it is possible under certain conditions for the carbon monoxide formed during the combustion to burn to carbon dioxide near the top section of the vessel, where catalyst concentration is low. When this occurs, very high temperatures result. In order to protect the regeneration equipment, use is made of emergency water sprays which are directed into the burning gas and, at times, into the catalyst bed. Flow of emergency spray water is controlled to the desired quantity while steps are taken to eliminate the factors leading to the upset condition.

The pressure at the outlet of the regenerator may be controlled automatically or manually. In case of automatic control, a pressure tap at the top of the regenerator is the control point for a valve positioner which adjusts the opening of a valve throttling the flue-gas flow.

For economical use of combustion air, it is desirable to maintain only a slight excess of oxygen in the regenerator flue gas. A visual record of oxygen content of this stream is obtained in many units by means of an oxygen recorder which samples at the regenerator outlet. As a further check upon operations, samples of flue gas are obtained regularly for determination of carbon dioxide, oxygen, carbon monoxide, and inserts by Orsat analysis.

Commercial Regeneration Operating Data

Table 4 summarizes some design and operating data for a commercial fluid cracking unit regenerator. It is to be emphasized that this tabulation illustrates operation of a specific installation and that the data are not typical for all units. For other units, in addition to variations in design details, the operating conditions may differ considerably from those shown. The unit under consideration is an installation designed for low catalyst-circulation rate between the reactor and regenerator and for moderate regeneration temperature, and has a recycle-catalyst cooler for control of regeneration heat balance.

TABLE 4

Regenerator Operation in a Fluid Catalytic Cracking Unit

Design Details:	
Vessel diameter	36'-6" I.D.
Shell length	40'-0"
Heads	Hemispherical
Top elevation	150'
Shell and heads	Carbon steel
Operating Conditions:	
Pressure at top of vessel	9 psig
Catalyst bed temperature	1025° F.
Entering air temperature	230° F.
Catalyst bed depth	18'
Catalyst bed density	20 lbs./cu. ft.
Spent catalyst charge	540 tons/hr.
Air supplied	220,000 lbs./hr.
Gas velocity at top of vessel	1.45 ft./sec.
Carbon on spent catalyst	1.80 weight-percent
Carbon on regenerated catalyst	0.35 weight-percent
Coke burned (8 percent hydrogen)	17,200 lbs./hr.
Flue gas analysis (dry)	
CO_2	12.0 volume-percent
O_2	2.0 volume-percent
CO	5.8 volume-percent
Inert	80.2 volume-percent
Steam produced	135,000 lbs./hr.

Smokeless Burning Of Refinery Vent Gases

A. K. BRUMBAUGH, JR., General Petroleum Corporation

MANY refineries which happen to be located in metropolitan areas face a severe problem in the disposal of waste light hydrocarbon materials, especially those gases and liquids which are discharged from safety relief valves and which consequently are produced in widely varying amounts not subject to direct control. This is particularly true of operations involving large gas-recovery, light-end fractionation, and alkylation units; for such equipment requires the holding in process vessels of considerable volumes of hydrocarbons. The special problems associated with materials in the propane-hexane range result from a combination of volatility at normal temperatures and vapor density appreciably greater than that of air; the high volatility means that discharged material may not be readily condensed, for example, by water washing, and the high-vapor density slows down dispersion by natural forces and encourages the formation of pockets of gas.

Most operators of this type of equipment have concluded that the only safe method of handling such vented material is burning. Early in 1946 General Petroleum Corporation instituted a program aimed at the development of equipment which would burn vent gases without objectionable smoke. The following discussion outlines the basis of this development and describes the installation recently completed.

As a first step a rather careful search of the literature was made but no reports of any approach to the problem were found. Without previous background, various methods of supplying and mixing air with the gas prior to ignition were studied with the result that the design of a venturi-type aspirating burner was attempted. Mechanical methods of aeration, for example by blowers, had to be ruled out because the volumes of gas to be handled were so great as to require equipment of prohibitive size. Information for the design of the experimental unit shown in Figure 1 was obtained from three sources given in the bibliography and this unit was set up for field tests.

It was found that isobutane vapors could be burned without smoke using a ratio of venturi throat area to gas orifice area of 4 to 1. When isobutane containing considerable amounts, possibly 20 percent, of unvaporized material was fed to the burner it was necessary to substitute a smaller gas orifice, thereby increasing this ratio to 16 to 1. With the 16-to-1 ratio the wet material burned without smoke, this despite the fact that liquid could be seen emerging from the burner mouth and entering the flame. Wet gas from the gas plant feed required a ratio of 8 to 1 for clean combustion. It was also determined that with the orifice ratio giving minimum satisfactory aeration the pressure upstream of the gas orifice could be reduced to ⅛ psig without flame popping back. Below this limit operation was unstable; eventually the flame would travel back through the venturi tube and burn at the gas orifice; it was discovered, however, that a pressure of about 1 psig would blow the flame through the tube and re-establish normal operation.

With the limitations of the venturi burner established, the application of such units to the refinery flare system was considered. As it was apparent that the permissible pressure at the gas orifice would control the size of the equipment, calculations were made to determine the effect of this back pressure on the relief valve system, with the result that 5 psig was chosen as the maximum figure. Because all the vessels tied into the burner system had design pressures of 100 psig or over, and the relief piping was adequately sized, it was found that for any reasonable assumption of emergency conditions the pressure rise in the protected vessel or vessels would be well within the margin of allowance set by the API-ASME Code. The effect of back pressure on the popping pressure of the relief valves could be ignored, as the greatest build-up at the downstream side of any valve was estimated at 15 psig while the set pressure was at least 25 psig below the design pressure of the vessel in question. It was also decided to design

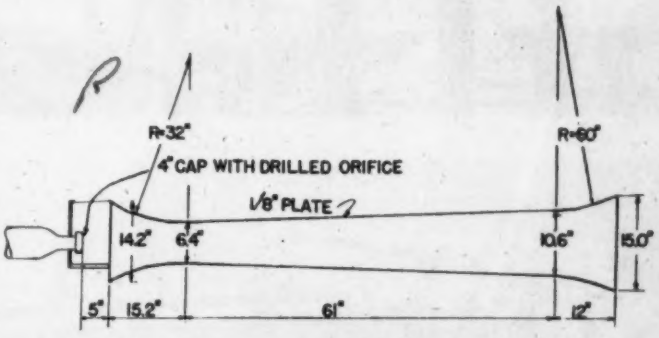


FIGURE 1
Experimental burner

the burner installation for a rate of 14,000,000 cubic feet daily, this seeming sufficient to handle any discharge resulting from emergencies.

With an operating pressure range of ¼ psig to 4 psig, a given gas orifice would have a range of volume burned of 4 to 1. As the volume to be handled could be expected to vary from zero to the maximum it was decided to employ several burners of varying sizes with some sort of pressure-responsive mechanism to cut them in successively. A conventional back-pressure control valve could not be used for this pur-

pose for as soon as the burner was turned on the increased flow would drop the header pressure with the result that the valve would quickly shut off again, resulting in a cyclic intermitten action. To meet this problem an adaptation of commercially available control valves was made as shown in Figure 2. With this mechanism adjustment, one pilot valve sets the opening of the main valve at any desired header pressure while the other pilot valve permits adjustment of the closing point to that pressure on the burner orifice which is the minimum for satisfactory operation.

A schedule of opening and closing pressures was set up for the series of burners, arranged so that the largest unit would be the last to cut in and the first to cut out. The size of the smallest burner orifice was next determined, being estimated from the surge capacity of the entire piping system and the normal amount of vapor leakage so that this burner would operate at fairly infrequent intervals to discharge accumulated material. The size of the next larger orifice was calculated from that of the smallest and the

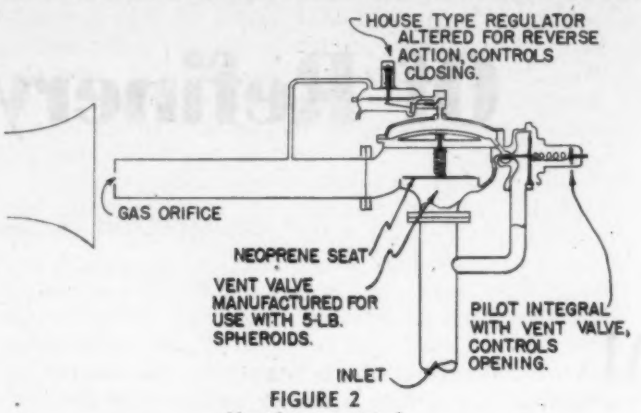
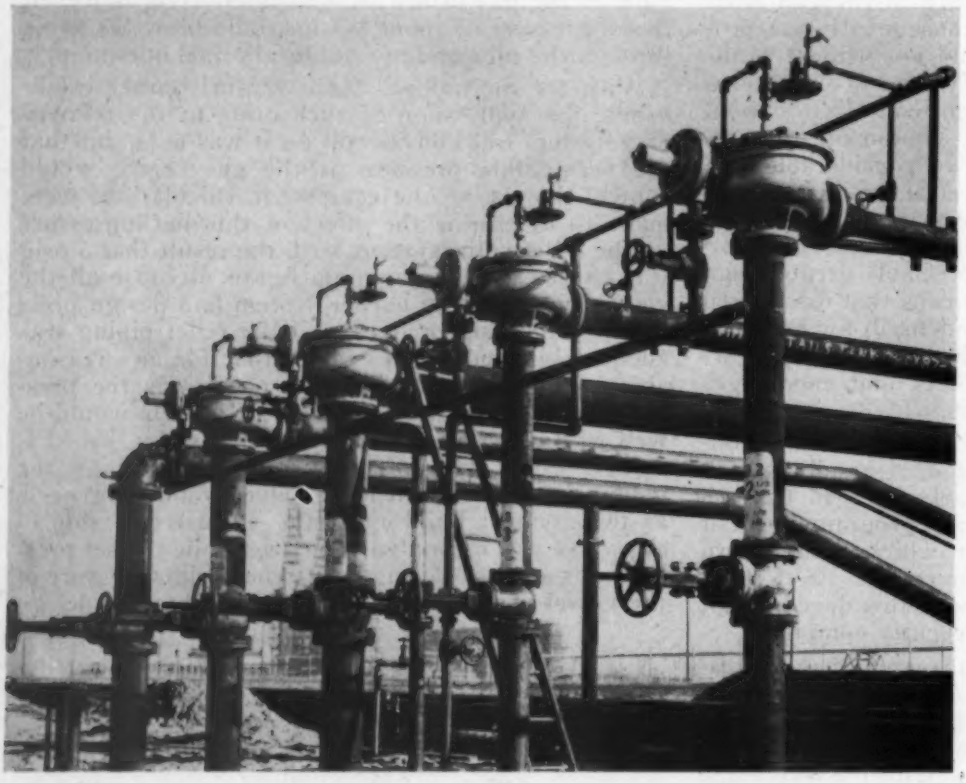
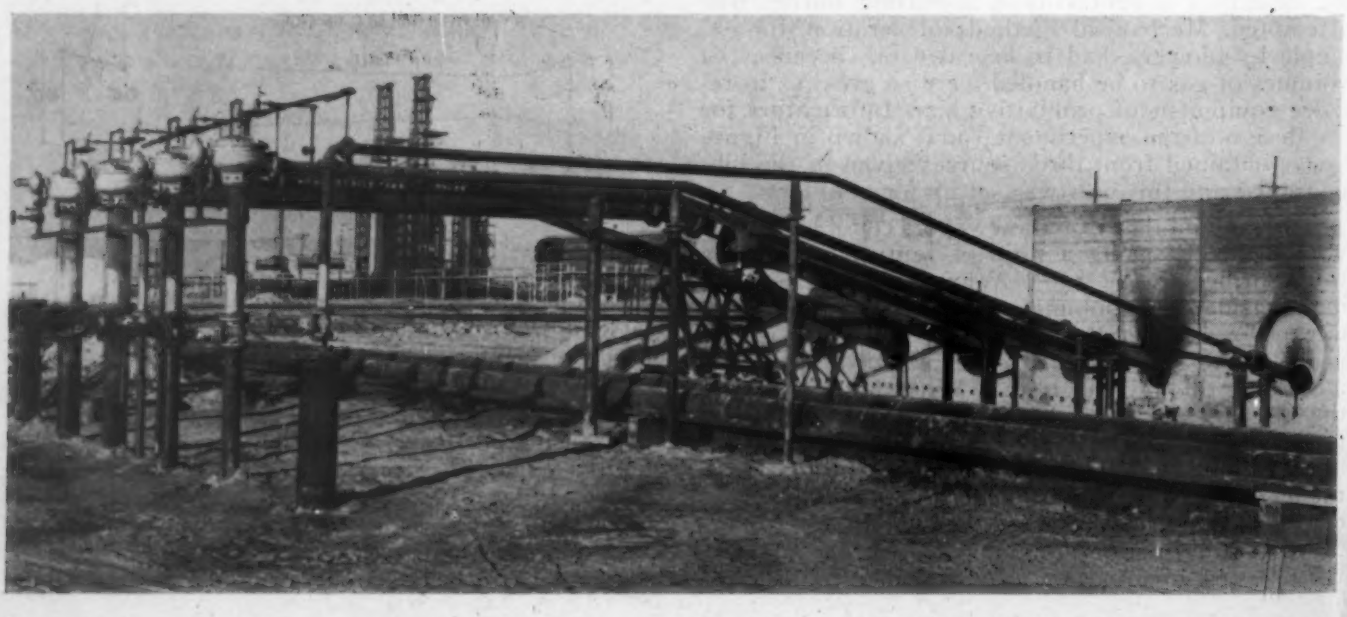

HOUSE TYPE REGULATOR ALTERED FOR REVERSE ACTION, CONTROLS CLOSING.
GAS ORIFICE
NEOPRENE SEAT
VENT VALVE MANUFACTURED FOR USE WITH 5-LB. SPHEROIDS.
PILOT INTEGRAL WITH VENT VALVE, CONTROLS OPENING.
INLET

FIGURE 2
Vent burner control



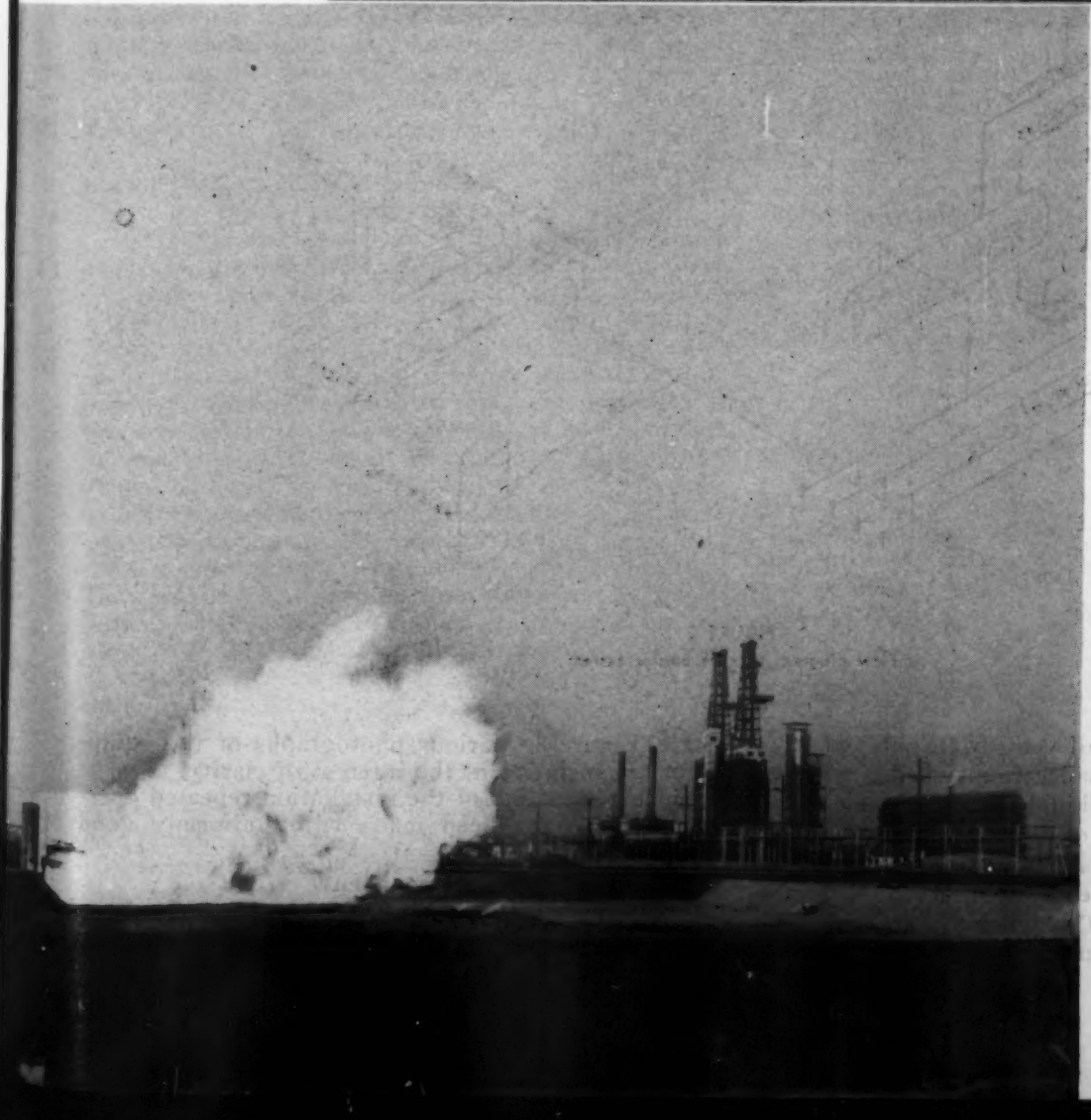
Left—Pilot-controlled fully automatic regulating valves.

Below—Regulator manifold and piping of burners.



BEFORE

Old method of burning emergency released light oil and gases which, due to lack of aeration, created clouds of black smoke.



AFTER

With new burner installation, General Petroleum Corporation's refinery at Torrance, California, handles emergency-released light oil and gas without smoke.

operating pressure schedule, being limited so that both burners at the cutout pressure of the second would burn only as much gas as the first burner alone at the cut-in pressure of the second. It will be seen that this method of graduation provides a stable operating condition for any gas flow in the range covered, thereby avoiding intermittent operation. Other gas orifices were determined, maintaining a similar relationship between the capacity of each additional burner and the total of those which would have previously cut in. Venturi-tube dimensions were established by direct proportion to the experimental unit which had given good results, selecting a venturi area to orifice area of about 8 to 1 as the most generally desirable. Sizes and pressures finally used after slight readjustments to conform to readily available material and after field tests together with other pertinent data are included in the following table.

Burner Number	Cut In Psi	Cut Out Psi	Gas Orifice Diameter Inches	Venturi Throat Dia. Inches	Ratio Throat Area to Orifice Area
1	2¼	¼	1.61	5	9.6
2	2¾	½	2.9	8	7.8
3	3¼	¾	4.03	11.5	8.1
4	3¾	1	7.0	18	6.6

To convey the vapors to the battery of burners all vent lines were combined in a 36-inch diameter underground header, each burner having an individual riser to its control valve. Provision was made for continuous drainage of condensate, all vents were pitched from the relief valve to the header from whence a sealed gravity drain conducted liquid to the refinery recovery system. Because lack of capacity or failure of the automatic valves to operate might impair the functioning of the entire relief system, a branch of the main header was led to a water seal where its open end was submerged beneath 10 feet 4 inches of water, resulting in assured release of vapors at header pressures over 4½ psig.

In order to avoid liquid hydrocarbons reaching the burners, as well as save valuable material for re-use, discharge lines from relief valves of all vessels operating full of liquid, and hand controlled drains from reflux drums and the like were segregated and led to a 10-foot diameter by 78-foot long accumulator which in turn was connected to the burner header. Liquid trapped in this vessel can be pumped back to the plant for recovery.

Experience with previous simple flare installations indicated that arrangement of the burner setting to provide satisfactory service life despite the tremendous evolution of heat during an emergency would require serious consideration. Experiments with the first unit showed that the aspirated air kept the venturi tube itself relatively cool, so it was thought that if provision were made to protect the burners from radiated heat and flame from adjacent units, fabricated steel venturis could be used. Thicknesses varied from ¼ inch for the smallest sizes to ½ inch for the largest. An air-cooled wall 10 feet high and 50 feet long was constructed, and the burners were installed flush with the surface through suitable ports. The wall consists of a 13-inch face of firebrick, a 2-inch air space, and an 8-inch thick reinforced concrete backing. Pipe nipples through the concrete along the bottom provide for convection circulation of air through the clearance space. All of the separate elements of this installation are coordinated in the flow chart, Figure 3. Various photographs of the equipment are included in the interest of clarity.

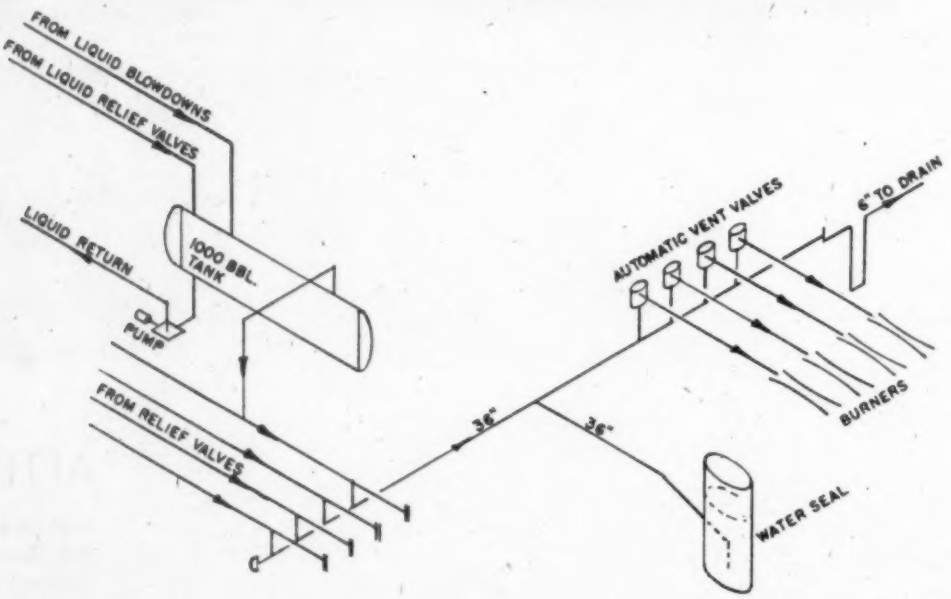


FIGURE 3
Flow diagram of vent burner system

Since completion of the installation repeated operating tests have been made with uniformly good results.

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Basic Petroleum Chemistry For Refinery Employes

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Chapter IX

POTENTIAL SOURCES OF LIQUID FUEL

THERE is growing interest on the part of business interests, research organizations and government agencies in the search for petroleum substitutes. This interest has been caused by the tremendous drain upon our petroleum resources during the past five years and the prediction by economists that the demands for petroleum products during the next five years will be greatly increased. Interest also has been stimulated by the rising costs of petroleum exploration and production, and the realization of the importance of an abundant supply of liquid fuel in time of war. A strong national defense requires an overwhelming abundance of many types of liquid fuel.

Even before the automobile became a popular means of conveyance, various authorities began to sound ominous warnings with reference to an early exhaustion of our available supply of crude oil. Perhaps the first "oil shortage" scare in this country was made for sales purposes, and it occurred even before the Drake well was drilled in 1859.

In 1874 Dr. Wrigley, State Geologist of Pennsylvania, wrote, "The present yearly output (of crude oil) is over 25 million barrels. . . . Some day the cheque will come back endorsed 'no funds' and we are approaching that day very fast."

In 1908 Dr. David Day, director of the United States Geological Survey, estimated maximum possible reserves in the United States to be about 23 billion barrels of oil. Since that time about 30 billion barrels have been consumed and known reserves as of January 1, 1947, stand at more than 21 billion barrels. Contemporary experts vary in estimates of the day when the check will be returned marked "no funds" from a low of 14 years to a high of 1000 years.[1]

With continued increasing consumption, eventual exhaustion of crude oil is inevitable; but today, contrary to the various warnings of early exhaustion, known reserves are greater than they have ever been before. Further, only about half of the probable oil-producing areas of the United States have been explored. Improved technology in synthesis, oil exploration and production is expected to expand reserves even more.

Government's Liquid Fuel Projects

Government interest in this matter has taken the form of Public Law No. 290, authorizing the Secretary of the Interior, through the United States Bureau of Mines, to construct and operate demonstration plants to provide synthetic liquid fuels from coal, lignite, oil shale, agricultural and forestry products, and $30,000,000 was appropriated for the purpose. Recently the Bureau of Mines asked that the appropriation for this project be doubled. The action of Congress in this matter may seem premature in view of known reserves of crude oil and natural gas, but, if for no other reason than its importance to national defense, it is justified.

The 11-member Senate oil-investigating committee, created two years ago to recommend a national oil policy, issued a statement recently to the effect that "bold steps" were in order to get synthetic fuel producion ready. Crude oil and source materials for synthetic fuels appear to be "capable of meeting all domestic petroleum requirements for many centuries," hence: "It is folly to be apprehensive that this nation is facing a shortage of liquid fuel." But, it asserted, in the event of war, with all imports cut off, we could not support a large scale campaign. "The reserves within our borders are more likely to constitute the citadel of our defense." It is urged that every possible step be taken to develop liquid-fuel resources, because, despite the atomic bomb, oil remains the key to military victory. The answer, declared the committee "is to be found in synthetic production."[2]

Natural Gasoline

It is estimated that known reserves of natural gas are sufficient to last only about 30 or 40 years at the present rate of consumption. However, new discoveries, improvements in conservation and intelligent utilization will extend the supply for a much longer time.

The total production of natural gas more than doubled between the years 1933 and 1943, and the present annual consumption is valued at more than half a billion dollars. Besides the many chemicals and chemical raw materials made from natural gas (see Chapter VIII, Table 10), there are produced about 280,000 barrels per day of light, volatile liquid fuel. This is called natural or casing-head gasoline, and is used for blending into automotive fuel. The first commercial process for the recovery of natural gasoline was by compression cooling. The natural gas is compressed by means of mechanical compressors and then cooled. This causes the hydrocarbon usable in gasoline to liquefy.

The compression-cooling method has been superseded to a great extent by the absorption method. This method is dependent upon the principle that the

hydrocarbons usable in liquid fuel are more easily dissolved in liquid petroleum fractions than are the lighter gaseous hydrocarbons. The gas stream is bubbled through absorption oil, which takes into solution the heavier hydrocarbons from the gas. The natural gasoline then is separated by means of distillation.

A third method of recovering liquid fuel from natural gas is known as the adsorption method. It involves the passing of natural gas through towers filled with solids, such as activated carbon, charcoal or silica gel, each of which is capable of retaining the hydrocarbons usable in gasoline in preference to the gaseous hydrocarbons. When the medium in the adsorption tower becomes saturated with gasoline, the gas is diverted to a second adsorption tower. The gasoline then is recovered from the adsorption medium by stripping with steam.

These three methods of gasoline recovery also have been used in refineries for the recovery of liquid fuel from refinery gas produced in cracking and distillation operations. High-pressure adsorption methods are more commonly used in gas-field and refinery operation today.

These methods depend merely upon changing the physical form of the desirable hydrocarbons from a gaseous to the liquid state. Research efforts now are directed chiefly toward changing the chemical structure of the natural-gas hydrocarbons.

The Synthine Process

The term "synthine" is a contraction of the words "synthetic gasoline," and is applied usually to a process which produces petroleum-like substances from the hydrogenation of carbon monoxide. The carbon monoxide may be produced from coal, coke, oil shale, natural gas, or possibly other carbonaceous materials. The hydrogen may be produced from water and natural gas. The raw materials, therefore, are abundant.

The original research upon which the synthine process is based had its beginning about 50 years ago in the experimental work done by the French chemist, Professor Sabatier of Toulouse. It remained for the Germans to attempt commercial application of the reaction.

Following World War I, two German chemists, Franz Fischer and Hans Tropsch of the Kaiser Wilhelm Institute, collaborated to make the Fischer-Tropsch synthesis of hydrocarbons from carbon monoxide and hydrogen, one of the most important in chemical history. Had the Fischer-Tropsch process been developed in 1939 to the extent that it is today, the mechanized German army would not have collapsed as early as it did, and might have rolled on to victory.

A rather small percentage of the liquid fuels produced in Germany during the war was produced by the Fischer-Tropsch process, and indications are that the cost of such fuels was much in excess of the cost of similar fuels from crude oil. It is quite well established, however, that German plants were clumsy and unnecessarily expensive. In the light of American engineering, research and development, several major oil companies are going ahead with the construction of synthetic gasoline plants with the expectation that the cost of the motor fuel produced will be at least competitive with the cost of a similar product from crude oil.[3]

Whether natural gas, coal, coke, shale, lignite or vegetation is used as raw material, the process first depends upon the production of a synthesis gas consisting of a mixture of carbon monoxide and hydrogen.[4, 5, 6, 7] Of the various possible sources, the production of the necessary synthesis gas from natural gas appears most economical at this time, and the commercial plants now being built will use natural gas instead of coal as raw material. Table 11 lists the comparative costs of Fischer-Tropsch gasoline from coal and gas, and modern refinery gasoline from crude oil at $1.20 per barrel and $2.00 per barrel.

Table 11

Costs per gallon of gasoline not including depreciation.

	Cents
Fischer-Tropsch gasoline from coal at $2.30 per ton	14.7
Fischer-Tropsch gasoline from natural gas at 5 cents per thousand cubic feet	6.0
Modern refinery gasoline from crude oil at $1.20 per bbl.	5.1
Modern refinery gasoline from crude oil at $2.00 per bbl.	8.3

These costs were estimated by R. P. Russell of Standard Oil Development Company in 1943.[8] Process improvements have lowered the estimated Fischer-Tropsch costs considerably. It is predicted that the cost of Fischer-Tropsch gasoline from the new plants now being built will be between 5 and 6 cents per gallon, while similar quality gasoline from coal would be between 7½ and 9½ cents with all costs being considered.

As previously stated, the Fischer-Tropsch process depends, first, upon the conversion of the raw material into a suitable synthesis gas. It might be possible to convert coal beds underground into the desired gas, thus eliminating mining, handling and some transportation costs. Russia has been converting coal underground into fuel gas for 12 years and a similar project is being developed in this country by Alabama Power Company in cooperation with the United States Bureau of Mines. The work will be carried on in a new mine at Gorgas, Alabama. Similar projects may be developed in connection with oil shale and lignite deposits[9]. Such projects, if successful, would have the effect of extending petroleum and natural gas reserves almost indefinitely. At the present rate of consumption, it is estimated that coal reserves are sufficient for 5000 years.

The production of a suitable synthesis gas from natural gas may be accomplished by one or several of the following reactions:

Reaction with steam according to the equation:

$$CH_4 + H_2O \rightarrow CO + 3H_2 \quad (1)$$

Reaction with carbon dioxide

$$CH_4 + CO_2 \rightarrow 2CO + 2H_2 \quad (2)$$

Reaction with oxygen

$$2CH_4 + O_2 \rightarrow 2CO + 4H_2 \quad (3)$$

Of six commercial methods of producing hydrogen for atomic hydrogen welding, manufacture of numerous chemicals, and the hydrogenation of petroleum and other oils, the reaction represented in the first equation above ranks second in importance. At least 17 plants having a total hydrogen-producing capacity of four billion cubic feet per month use this process. Modifications of the process are applicable to the synthine gasoline process.

Synthesis gas from coal is an adaptation of the old water-gas reaction between superheated steam and coke. Coke is the residue from the distillation of coal in retorts, petroleum in coke stills or more usually from coal by-product coke ovens. The reaction is as follows:

$$C\ (coke) + H_2O \rightarrow CO + H_2 \quad (4)$$

By the variation of temperature, catalysts and the

ratio of one reactant to the other, the ratios of carbon monoxide to hydrogen may be controlled in each of the above reactions to yield a synthesis gas best suited to synthine gasoline production.

Regardless of the source, synthesis gas must be practically free of sulfur compounds. About one grain per 1000 cubic feet is all that can be tolerated with most catalysts without serious poisoning of the catalysts. The natural gas may be treated prior to the synthesis gas reaction by gas purification methods such as those discussed in Chapter VII. A number of treating processes for synthesis gas have been proposed.

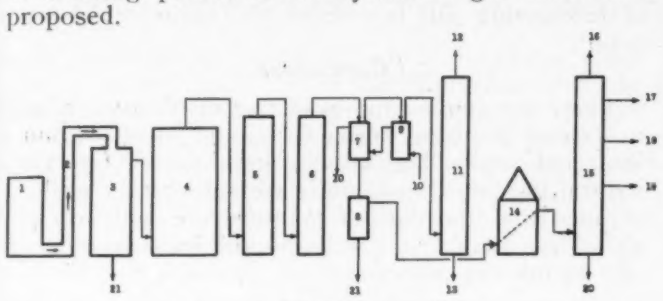

FIGURE 21

Synthine Process with coal as charge.

1. Coal supply; 2 Coal elevator; 3 Synthesis gas producer; 4 Gas holder; 5 Gas purification unit; 6 Synthesis reactor; 7 Condenser; 8 Water separator; 9 Cooler; 10 Cooling water; 11 Stabilizer; 12 Propane and Butane; 13 Stabilized gasoline; 14 Tube heater; 15 Fractionating tower; 16 Gas; 17 Naphtha; 18 Kerosine; 19 Furnace and diesel fuel; 20 Wax and heavy oil; 21 Waste.

Following the purification, the next step in the synthine process is the catalytic synthesis of the synthesis gas to hydrocarbon and water according to reactions of which the following are typical:

$$nCO + (2n+1)\,H_2 \rightarrow Cn\,H_{(2n+2)} + nH_2O \qquad (5)$$

Where n = 6 for example:

$$6CO + 13H_2 \rightarrow C_6H_{14} + 6H_2O$$

Hexane

$$nCO + 2nH_2 \rightarrow Cn\,H_{2n} + nH_2O \qquad (6)$$

Where n = 6 for example:

$$6CO + 12H_2 \rightarrow C_6H_{12} + 6H_2O$$

Hexene

Reactions such as that represented in equation (5) above are favored by a relatively high ratio of hydrogen to carbon monoxide in the synthesis gas. The hydrogen type formula, $CnH_{(2n+2)}$ will be recognized as the type formula for saturated or paraffin hydrocarbons, while CnH_{2n} is the type formula for unsaturates or monolefins. The latter reaction is favored by low hydrogen-carbon monoxide ratios and catalysts which have less hydrogenating ability[5].

Following the synthesis reaction, the reaction products are passed through a condenser. The condensate is heated in a tube heater and separated into fractions. The uncondensed portion through the condenser, containing the gas and gasoline, is debutanized in a conventional stabilizer column to the desired gasoline vapor pressure. Figures 21 and 22 are simplified flow diagrams representing gasoline production from natural gas and coal by the synthine process. Figure 21 represents a process in which reaction (3) (above) is used for the production of synthesis gas.

The primary products from the synthine process are as diversified in their known and probable uses as are the various products from crude oil. In Germany, detergents, fatty acids, edible fats, lubricants, solvents and chemicals, in addition to fuels for automobiles, planes and diesel engines, have been made by the synthine process applied to coal. Diesel fuels with high cetane number can be made by the synthine process.[16]

Hydrogenation of Coal

A very large percentage of the fuel which launched and nearly won the war for Germany was produced by the hydrogenation of coal. Production of gasoline from coal by the direct hydrogenation route involves three major steps: First, the pulverizing and mixing of the coal with an oil carrier; second, the hydrogenation of the heavy oil and coal slurry, and finally the separation of the hydrogenated products from the slurry. The slurry consists of about equal parts of clean pulverized coal and heavy tar-oil. It is caused to react with hydrogen in vertical alloy-steel tubes heated to about 850° F. under pressures up to 4500 psi. About 20 percent boils between 400° and 626° F. This "medium oil," as it is called, may be further hydrogenated or cracked and fractionated to yield more gasoline, diesel fuel, fuel oil, tar bases, tar acids, and other substances.

The catalysts, which may consist of iodoform, stannous sulfide, molybdenum trioxide, molybdenum disulfide and other metalic oxides or sulfides, are suspended in the slurry in amounts ranging from 0.05 to 0.5 percent. This catalyst is recovered from the oil after it passes through the reactors by means of centrifuges or pressure filtration.

Yields obtained on experimental work by the United States Bureau of Mines indicate 7.5 to 12 gallons of gasoline per ton of coal. This yield is based on the coal used for the hydrogenation and that coal required in the process for heat and hydrogen production.

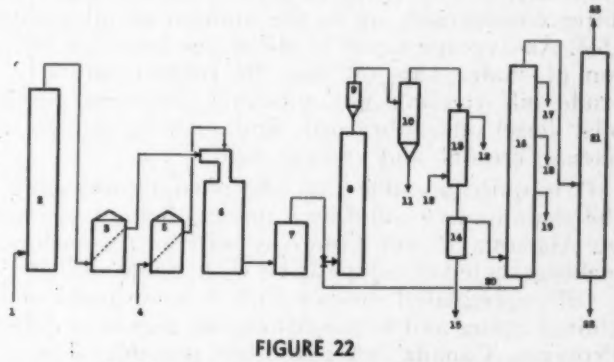

FIGURE 22

Synthine Process with natural gas as charge.

1 Natural gas charge; 2 Gas purification unit; 3 Gas tube heater; 4 Oxygen; 5 Oxygen tube heater; 6 Synthesis gas producer; 7 Waste heat boiler; 8 Fluid catalyst reactor; 9 Cyclone separator; 10 Wax separator; 11 Wax and heavy oil; 12 Cooling water; 13 Condenser; 14 Water Separator; 15 Waste; 16 Fractionating tower; 17 Naphtha; 18 Kerosine; 19 Furnace and diesel fuel; 20 Recycle; 21 Stabilizer; 22 Stabilized gasoline; 23 Gas.

The cost per gallon of gasoline in the work carried on so far has been between 15 and 18 cents. It should be pointed out, however, that with large-scale units and improved methods this cost could be considerably reduced, but seemingly not sufficiently to compete with the synthine process. The installation costs for hydrogenation plants are high because of the high temperatures and pressures used in the process.

Shale Oil

It is estimated that there are over 100 billion barrels of shale oil locked up in the shale deposits of the United States. This is an amount about five times greater than the known reserves of crude oil. Large quantities of oil shale are found in Colorado, Utah, Wyoming, Kentucky, Ohio and Indiana. Lesser amounts are found in other states. The shale richest in oil is found in Garfield County, Colorado.

Unfortunately there are very few scientific data

of recent origin available concerning the chemical composition or the methods of refining oils recovered from shale. Experimental work conducted by the Bureau of Mines has been along lines suggested by experience in Australia, Scotland and Esthonia. These have been on a rather small scale as judged by American standards. Work now is going on in some state universities which promises to add considerably to the fund of knowledge on this subject, particularly as it relates to modern petroleum technology. The work authorized by Congress in Public Law No. 290 provides for the accumulation of data by research in oil shale mining, retorting and refining. It also provides for the construction of demonstration plants of sufficient size to enable commercial-scale operations.

As a part of the Bureau of Mines' efforts in the shale-oil project, it has prepared a compilation and analysis of all patents concerned with the treatment of shale oil and its products. Some 2000 patents have been studied and they cover all phases of the subject of mining and retorting. A major problem in the retorting of shale is its coking tendencies when heat is applied. A recent patent development is concerned with the adaptation of the fluid catalyst technique to shale-oil retorting.

Oil, as such, does not exist in oil shale; instead, there exists a complex organic substance called "kerogen." This kerogen may be removed from the shale by heating in retorts to yield a petroleum-like oil. It may be removed also by extraction with organic solvents, or by hydrogenation. American shales differ considerably as to the amount of oil recoverable. An average figure is about one barrel of oil per ton of shale. This oil may be refined similarly to crude oil, yielding gas, gasoline, kerosine, furnace oils, diesel oils, lubricants, and such by-products as phenol, cresols, and cresylic acids.

It is quite possible that the partial processing of the shale underground by a process being considered by Alabama Power Company will be the means of making shale oil competitive with crude oil.[9, 10, 11]

Oil-impregnated sands found in some parts of the United States and in the Athabaska region of Alberta Province, Canada, offer another possible source of liquid fuels. Commercial methods of extracting the oil from these sands have been developed in Canada. The sand oil is said to be of better quality than many American crudes, because it has not been subjected to high pressure and temperature far beneath the earth's surface. The oil-impregnated sands of Alberta have been estimated to contain up to 250 billion barrels of oil[12].

Liquid Fuels from Agricultural Wastes

There has been much propaganda and some attempted legislation designed to support the use of alcohol in motor-gasoline blends. The chief purpose of such attempts has been to create a market for excess production of certain farm crops.

Unfortunately, while ethyl alcohol has a high octane blending value, its cost to date has been far too high to justify its use as a motor fuel—even in gasoline blends containing 10 percent or less alcohol. Further, alcohol has only about two-thirds the heating value per pound as compared to the usual hydrocarbon fuels, and it is relatively insoluble in gasoline unless it is anhydrous. Dilute blends have a tendency to separate into two phases under ordinary storage conditions in the presence of water or high humidity.

The demand for ethyl alcohol has been increasing rapidly because of its diversified uses in the manufacture of organic chemicals. This fact results in keeping the price high[13].

As a part of the government's liquid-fuel-research program, the Department of Agriculture has a semi-commercial plant at Peoria, Illinois, which will establish cost data concerning the manufacture of alcohol, butanol, acetone, isopropyl alcohol and other by-products from corn cobs, and other farm wastes. The plant will have a capacity of 500 gallons of alcohol per day. Furfural, a chemical used in solvent refining of lubricating oil, is one of the valuable by-products[14].

Conclusion

There are ample sources of raw materials available to extend probable needs for liquid fuels beyond a thousand years. The known methods of converting natural gas into liquid fuels are sufficiently well developed that the cost of manufacture will be equal to or less than the cost of liquid fuels from crude oil—at present prices.

The manufacturing costs of liquid fuels from coal, lignite, oil shale or agricultural wastes are high, resulting in commodity costs of from 5 to 10 cents per gallon greater than prevailing prices of liquid fuels from crude oil.

A recent Associated Press release quoting the annual report of Director R. R. Sayer of the Bureau of Mines said that synthetic gasoline would be on the market in this country "within a few years," and that the cost of production from coal and oil shale is now between 7½ and 9½ cents per gallon[15].

When these sources of liquid fuel begin to be used more extensively, certain refinery techniques will have to be changed. Their use in supplementing crude oil supply will have an important effect upon the refinery of the future. Just what effect they will have can not yet be accurately anticipated, but certain trends pointing to future refinery practice can be predicted with a fair degree of success. Some of these predictions make up the subject matter of the tenth and final chapter of this series, "The Refinery of the Future," to appear in an early issue of PETROLEUM REFINER.

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Roof Steam Coils Eliminate "Cloud" in Lubricating Oil Storage

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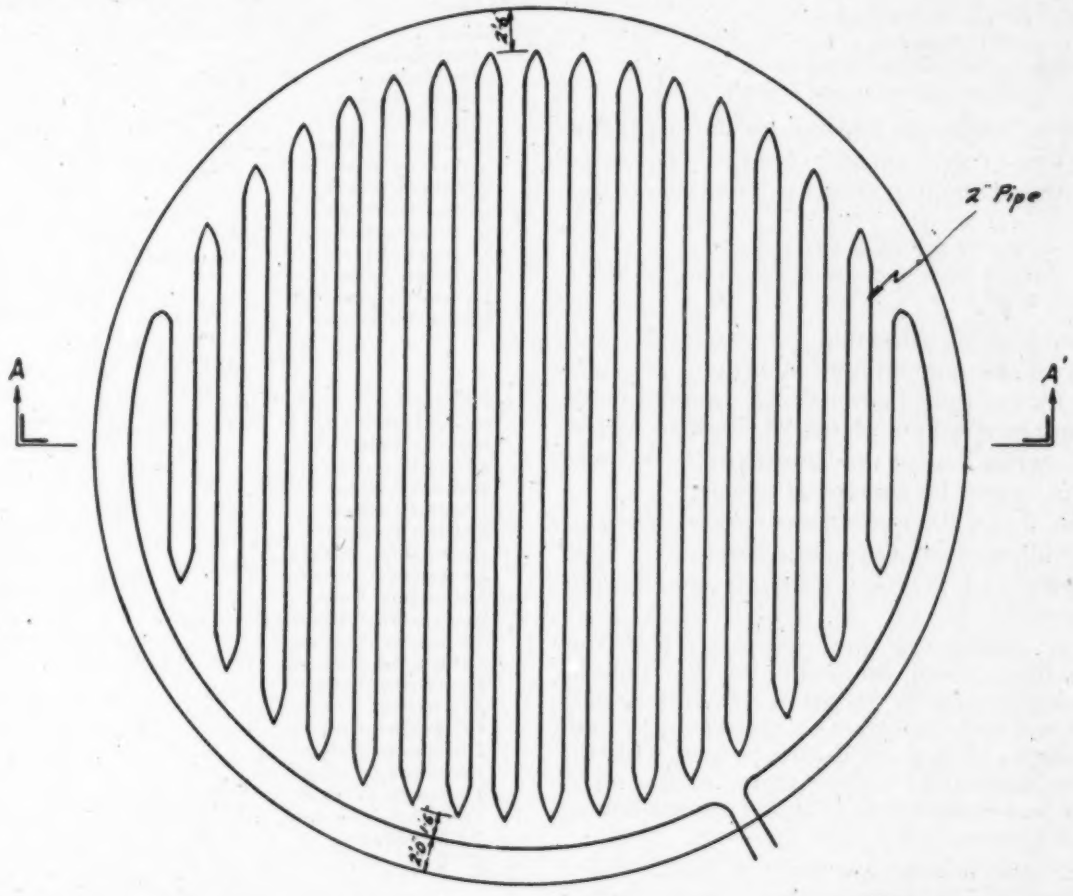
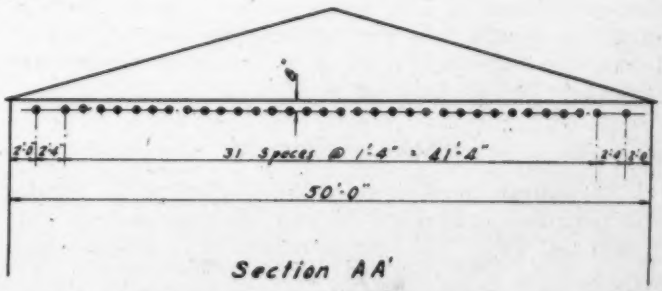
FINISHED lubricating oils may develop a hazy appearance or "cloud" in storage. This cloud may be caused by the presence of minute quantities of high-melting-point waxes or by moisture. While the wax cloud can be eliminated by taking the necessary precautions in the course of refining, the cloud arising from the presence of moisture may develop in storage because of condensation of water vapor from the atmosphere. This may be avoided by preventing the possibility of such condensation occurring while the oil is in storage.

Moisture will condense when the temperature of the air falls below the dew point, which may easily occur in regions where the humidity is high. Furthermore, tank breathing in certain seasons may be considerable, thus accounting for accumulation of water vapors inside the tank. In order to prevent condensation of this moisture, the air in the tank must be kept above the dew point and this can be accomplished by heating the air space within the tank. This also reduces the amount of breathing.

The problem was solved by installing steam coils in the tank roof. The accompanying diagram illustrates this steam-coil arrangement in one of the storage tanks.

The coils are hair-bend type suspended from the roof structurals with quarter-inch steel U rods. Other details of installation are given in the diagram. Steam to these coils is supplied from yard lines and the coil return line is trapped. Hand control is quite satisfactory for regulating the amount of steam admitted to the coils.





The above described system has been in operation for a number of years and is of proven practical value. No clouds, due to moisture, ever develop in these tanks.

Nomograph for Normal Boiling Points of Hydrocarbons

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EGLOFF, Sherman & Dull[1] showed that the normal boiling points of 31 homologous series of aliphatic hydrocarbons could be correlated by means of the equation

$$T = 745.42 \log (n + 4.4) + k$$

where T = normal boiling point °C.
n = number of carbon atoms in the molecule
k = a constant characteristic of each series.

Later, Corbin, Alexander and Egloff[2] showed that a similar but more general equation held for the correlation of the normal boiling points of aromatic hydrocarbons

$$T = A \log (n + 4.4) + k$$

where T & n are as before, and a & k are further constants characteristic of each series.

These equations are presented in nomograph form, the accuracy of the nomograph being approximately the same as that of the equations, the observed mean deviation for 143 numbers of the 31 aliphate series[1] being 0.7° C. while that of the 9 aromatic series[2] was not given but would be somewhat greater.

In order to obtain the optimum accuracy the equations for the aliphatic and aromatic series were handled separately and the two resultant nomographs superimposed.

To use the nomograph the appropriate point for the series is first chosen, the points for the aliphatic series all lying on the inscribed scale while points for the aromatic series appear as a cluster in the middle of the chart. This point is then joined with the point for the number of carbon atoms in the molecule and the line extended to give the normal boiling point of the hydrocarbon.

Conversely the nomograph may be used to find the component of any series within a given boiling range by connecting the temperature and the series point, the number of carbon atoms in the hydrocarbon then being obtained directly.

Acknowledgment

The author wishes to acknowledge with thanks the permission granted by the Management of National Oil Pty., Ltd., for permission to publish this paper.

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KEY
ALIPHATIC SERIES
ALKANES

Series	Scale Point
Alkane	0.0
2-Methylalkane	—8.2
3-Methylalkane	—6.6
4-Methylalkane	—8.3
3-Ethylalkane	—6.7
2,2-Dimethylalkane	—19.0
2,3-Dimethylalkane	—9.6
2,4-Dimethylalkane	—15.0
2,5-Dimethylalkane	—15.6
2,6-Dimethylalkane	—14.8
2,7-Dimethylalkane	—14.4
3,3-Dimethylalkane	—13.8
3,6-Dimethylalkane	—14.2
7,8-Dimethylalkane	—18.3

ALKENES

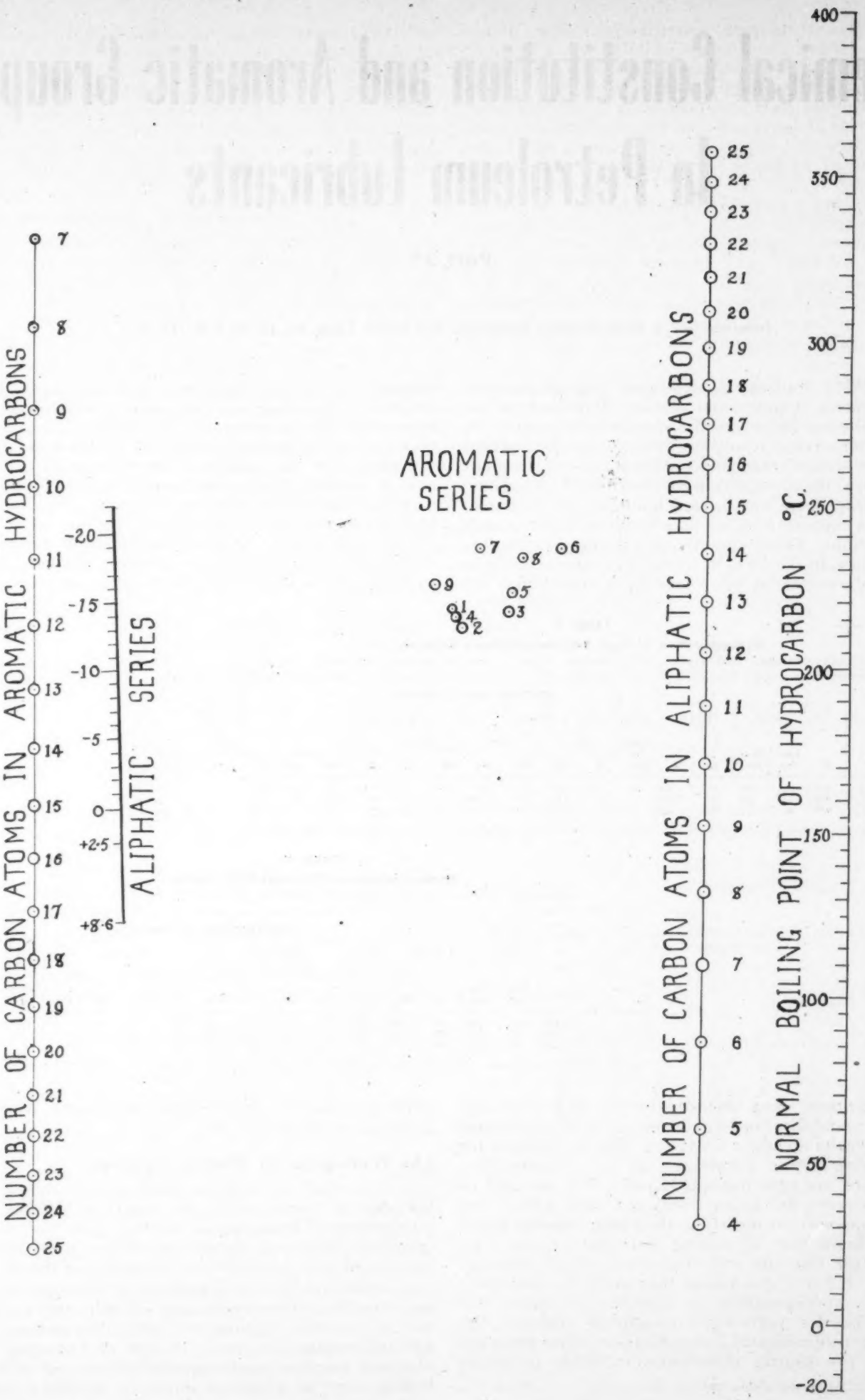
1-Alkene	—5.6
cis-2-Alkene	0.0
trans-2-Alkene	—0.3
2-Methyl-1-Alkene	—6.5
3-Methyl-1-alkene	—14.8
4-Methyl-1-alkene	—11.9
2-Methyl-2-alkene	—2.9
2,3-Dimethyl-1-alkene	—14.8
2,4-Dimethyl-1-alkene	—15.3
2,5-Dimethyl-1-alkene	—14.1
2,6-Dimethyl-1-alkene	—15.5
3,3-Dimethyl-1-alkene	—21.4
3,4-Dimethyl-1-alkene	—12.4
3,5-Dimethyl-1-alkene	—20.2
3,7-Dimethyl-1-alkene	—20.2
4,4-Dimethyl-1-alkene	—19.1
2,3-Dimethyl-2-alkene	—5.8
2,4-Dimethyl-2-alkene	—16
2,5-Dimethyl-2-alkene	—13.4
2,6-Dimethyl-2-alkene	—11.6
1,2-Alkadiene	+8.6
C=C-(C)$_x$-C=C.	—8.5

ALKYNE

1-Alkyne	+2.5

KEY
AROMATIC SERIES

Series	Point No.
1 phenyl alkane	1
2 phenyl alkane	2
1 phenyl 1 alkene	3
1 phenyl (n-1) alkene	4
1 phenyl 1 alkyne	5
1.1 diphenyl alkane	6
1.n. diphenyl alkane	7
1.1 diphenyl alkene	8
phenyl cyclane	9


NUMBER OF CARBON ATOMS IN AROMATIC HYDROCARBONS
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
-20
-15
-10
-5
0
+2·5
+8·6
ALIPHATIC SERIES
AROMATIC SERIES
7
8
6
9
5
1
4
3
2
NUMBER OF CARBON ATOMS IN ALIPHATIC HYDROCARBONS
25
24
23
22
21
20
19
18
17
16
15
14
13
12
11
10
9
8
7
6
5
4
NORMAL BOILING POINT OF HYDROCARBON °C.
400
350
300
250
200
150
100
50
0
-20

Chemical Constitution and Aromatic Groups In Petroleum Lubricants

Part 2*

C. ZERBE and K. FALKENS

Translated by E. J. Barth, Petroleum Technologist from Brennst. Chem. Vol. 16, No. 9 (P. 161, etc.)

IN PART I of our contribution[1] on the aromatic constituents of petroleum oils we described the results showing the chemical constituents present in the liquid SO_2 extract resulting from extracting a spindle-oil. We showed that the constituents were aromatic in nature; the aromatics were distributed throughout the extract from low-to high-boiling-point hydrocarbons as separated by us in a series of high-vacuum distillations. These cuts showed various viscosities. These oils, in the form of fractions, cannot exactly be used as commercial lubricants due to their high sulphur content, steep viscosity curves (low viscosity-index) instability, and a great tendency to form resins and asphalts on slight oxidation. The possibilities for employing these E-extracts, as is, commercially, therefore, are very limited. Usually they are sold as fuel oils—not due to any lubricant value which they might possess but merely to their high calorific value.

We knew that by adding hydrogen we could increase the viscosity and viscosity-index of these extracts[2]; but it is also known that unstable compounds can, by hydrogenation, be converted to stable material besides reducing the sulphur content. We therefore investigated hydrogenation under pressure. One can control this operation either to obtain complete cracking or complete non-cracking hydrogenation, depending on the working conditions of temperature, time, nature of the catalyst, etc. In the case of straight hydrogenation one would expect improvements in the quality of the lubricants. In the case of cracking-hydrogenation one expects to obtain valuable low-boiling-point hydrocarbons suitable for high-grade motor-fuel production. In our work we sought especially to preserve the lubricant nature of the oil and not to destroy any inherent viscosity. The hydrogenations therefore were carried out with and without catalysts in temperature ranges wherein splitting would not occur.

TABLE 1

Hydrogenation of Raw E-Extract Without Catalyst

(Quantity used: 200 gm.; preheating 1 hr.; catalyst: 10 gm.; time at highest temperature: 2 hrs.)

No.	°C.	PRESS. Init. (Atm.)	PRESS. Max. (Atm.)	YIELD %	YIELD Dis.	H_2 Press. Used Up (atm.)	DISTRIBUTION OF PRODUCT At 13 m.m. —150 C. %	—150 C. Dis.	—200 C. %	—200 C. Dis.	At 4 m.m. —200 C. %	—200 C. Dis.	—250 C. %	—250 C. Dis.	Res.+ Loss	% S.
1	430	60	180	91	0.984	10	22	0.81	39	1.0	14	1.01	18	1.01	6.0	2.0
2	470	60	170	90	0.982	15	30	0.85	35	0.960	19	1.00	18	1.01	8.0	1.7
3	470	60	170	90	0.980	15	28	0.86	34	0.960	19	1.00	11	1.01	8.0	1.7

TABLE 2

Hydrogenation of E-Extract With Iodine.

(Quantity: 200 gm.; preheating 2 hrs,; time at maximum temperature: 2 hrs.)

No.	°C.	PRESS. Init. (Atm.)	PRESS. Max. (Atm.)	YIELD %	YIELD Dis.	H_2 Press. Used Up (atm.)	DISTRIBUTION OF PRODUCT At 13 m.m. —150 C. %	—150 C. Dis.	—200 C. %	—200 C. Dis.	At 4 m.m. —200 C. %	—200 C. Dis.	—250 C. %	—250 C. Dis.	Res.+ Loss	% S.
1	400	60	140	90	0.980	18	20	0.82	23	1.0	18	1.0	33	..	6.0	2.2
2	430	60	170	90	0.970	20	30	0.85	34	0.96	20	1.0	8	..	8.2	1.8
3	470	60	160	90	0.970	30	35	0.84	36	0.93	14	1.0	9	..	7.1	1.8

(A) Hydrogenation Without Catalysts.

In this series we used no catalyst and studied the behavior of E-extract in the range of 350-400° C. temperature. These tests showed at once that under these conditions we obtained no addition of hydrogen and no valuable improvement in quality of the oil.

In order to obtain any addition of hydrogen at all one must employ temperatures of over 400° C., and here appreciable cracking will occur. For instance, at 430° C. despite consuming 10 atm. of hydrogen, we obtained reaction products which consisted of low-boiling-point aromatics as shown by specific gravity,

* Part 1, Petroleum Refiner **26**; 101 (Feb., 1947).

refractive index, and boiling-point range. (Text No. 1); at 470° C. the consumption of hydrogen is somewhat greater (15 atm.), and cracking increases (Test No. 2). Addition of a sulphur-combining catalyst to remove large amounts of hydrogen sulphide is without effect (Test No. 3); the sulphur content of the hydrogenated product is 1.8 percent on the average, a reduction of about 50 percent over that of the original raw material.

(B) Hydrogenation With Catalysts.

By the use of catalysts substantial addition of hydrogen takes place. As catalysts we employed elementary iron and the sulphides of molybdenum, and investigated these thoroughly since they showed to be the most promising in much of our previous work.

(1) Iodine as catalyst. (See Table 2)

Using iodine one obtains at 350° C. a substantial addition of hydrogen as compared to runs at 350° C. without catalyst; this temperature, however, still does not secure a sufficient improvement in the oils—that is, the production of high-grade lubricants. For that purpose we had to employ temperatures of over 400° C. which was high enough to cause cracking, and a good portion of high-viscosity material was destroyed.

As to sulphur content, the reaction products show no difference from the reaction products made without the catalyst.

(2) Molybdenum as Catalyst. (See Table 3)

Sulphides of molybdenum showed much better catalytic activity than iodine. Due to the high sulphur content of the raw stocks we could employ molybdic acid instead of molybdenum sulphide; the acid combines, during the hydrogenation, with the sulphur which splits off, forming a molybdenum sulphide in the reaction mixture. This form of the sulphide seems to be quite superior to commercial MoS_2.

The activation of the catalyst by sulphur commences at about 300° C. and is completed quite rapidly.

Using molybdenum catalysts one notes appreciable splitting at 400° C., which is noticeable by a very sudden rise in pressure due to formation of considerable methane-type hydrocarbons in the reaction-gases. In order to prevent injury to the apparatus alkali-flux was employed to combine with the hydrogen sulphide. This material which is added does not affect the catalyst. Silica gel was found to be the best supporter for the molybdenum.

When one hydrogenates one of the primary distillates from the distillation instead of the E-extract itself, there is a definite addition of hydrogen at 360° C., which far surpasses the quantity and results obtained at 400° C. The hydro-products then are far more stable, contain very much less low-boiling-point benzole derivatives, and the higher-boiling-point fractions, especially, show excellent high viscosity. The sulphur contents could not, however, be further reduced than shown in Tables 1 and 2. This observation agrees with results Roberti[3] found in the case of Ragusa shale-oil.

After our runs with the total extract had indicated optimum conditions for hydrogen-addition, we proceeded with and enlarged the number of experiments with the individual fractions. (See Table 4.)

As shown by the results in Table 4, one will note in the case of these individual fractions that far-reaching splitting is occurring at temperatures over 400° C. into benzole-like low-boiling-point hydrocarbons.

Maintaining the reaction-temperature constant at not over 350° C., other conditions being equal, the hydrogenation will predominate. With increase in boiling-point of the cut, and simultaneous rise in specific gravity of the reaction product there is a diminuation of hydrogen-absorption for each cut. The higher-boiling-point fractions of the E-extract show, therefore, a more saturated nature and are more difficult to hydrogenate.

Parallel with this resistance to hydrogenation there is the stability of the molecule itself. Following from

TABLE 3

Hydrogenation of Raw E-Extract in Presence of Molybdenum.*

(Quantity: 200 gms.; preheat time: 2 hrs.; time at maximum temp.: 2 hrs.)

No.	°C.	PRESS. Init. (Atm.)	PRESS. Max. (Atm.)	YIELD %	YIELD Dis.	H_2 Press. Used Up (atm.)	DISTRIBUTION OF PRODUCT At 13 m.m. —150 C. %	—150 C. Dis.	—200 C. %	—200 C. Dis.	At 4 m.m. —200 C. %	—200 C. Dis.	—250 C. %	—250 C. Dis.	Res.+ Loss	% S.
1	360	60	140	92	0.970	30	20	0.82	23	1.0	21	1.0	30	1.01	6.0	2.1
2	400	60	150	90	0.968	40	16	0.85	45	0.94	15	0.99	16	1.00	8.0	1.9
3	430	60	150	90	0.968	60	18	0.85	50	0.93	18	0.96	6	1.00	8.0	1.8
4*	360	60	140	91	0.970	50	21	0.83	26	1.0	19	1.00	28	1.01	6.0	1.9

* Using molybdenum sulphide.

TABLE 4

Hydrogenation of Fractions of E-Extract with Molybdenum and 10 gm. Alkali-Flux.

(Quantity: 200 gm.; preheat time: 1 hr.; time at maximum temperature: 2 hrs.)

Cut	Test No.	Max. °C.	PRESS. (Atm.) Initial	PRESS. (Atm.) Max.	H_2 Used Up (Atm.)	YIELD %	YIELD Dis.	Res. + Loss	%—S.
1	1	420	80	190	80	89	0.920	7.0	1.3
1	2	350	80	170	50	90	0.940	6.0	2.0
2	3	400	80	170	75	86	0.920	3.0	1.6
4	4	350	80	170	45	90	0.960	8.0	2.0
4	5	350	80	170	40	90	0.980	5.0	2.1
5	6	350	80	170	35	91	0.981	6.0	2.3
6	7	350	80	190	35	90	0.986	5.0	2.5
9	8	....	....	....	30	90	0.986	4.0	2.6

Cut	Test No.	DISTRIBUTION OF PRODUCT At 13 m.m. —150 C. %	—150 C. Dis.	—150 C. Nd_{20}	—200 C. %	—200 C. Dis.	—200 C. Nd_{20}	At 4 m.m. —200 C. %	—200 C. Dis.	—200 C. Nd_{20}	—250 C. %	—250 C. Dis.
1	1	67	0.88	1.496	23	0.929	1.520	3	1.01	1.534	....	....
1	2	12	0.89	1.496	18	0.929	1.520	36	1.00	....	28.0	1.01
2	3	60	0.88	1.497	21	0.930	1.502	16	1.00	....	....	....
4	4	10	0.89	1.496	16	0.930	1.550	40	1.00	....	26	1.01
4	5	10	0.89	1.497	15	0.930	....	41	1.00	....	29	1.01
5	6	8	0.86	1.495	15	0.929	....	40	1.00	....	31	1.01
6	7	5	0.89	1.496	15	0.940	....	50	0.99	....	25	1.01
9	8	2	0.88	1.497	12	0.940	....	51	1.00	....	31	1.02

that, one notes a lesser and lesser tendency toward cracking in the case of the higher fractions. The autoclave products contain, therefore, much less low-boiling-point hydrocarbons and there is less methane in the reaction gases. The higher-boiling-point sulphur compounds also are much more stable, as shown by the increasing sulphur contents of the higher cuts as found in the autoclave contents.

The loss of about 10 percent in the whole operation is due to cracking. The especially high content of methane-hydrocarbons in the reaction gases, which amounts to about 50 percent after the first period of heating, and about 20 percent after the second heating period, points to the fact that a considerable amount of aliphatic side-chain material is split off. This had been followed and proven by optical measurements. Nevertheless, one obtains in the case of high-boiling-point portions of the hydro-product oils whose viscosities are much heavier than the corresponding boiling-point cuts of the raw crude material. We cannot ascribe at this time what this is due to—perhaps to hydrogenation in presence of sulphur, or to stabilization and polymerizations which proceed due to the polymerization effects of sulphur itself. Compounds which diminish viscosity or which thin out heavy oils also probably are destroyed by the cracking which takes place.

The hydro-products show, in comparison to the true paraffinic petroleum oils, quite a decrease of viscosity with rise in temperature. This decrease is, however, much less pronounced than in the case of the raw crude material. The oils are completely stable, do not darken on standing, and have lost all tendency to form resin or pitchy matter on heating.

Summary

The behavior of an E-extract of an asphalt base spindle-oil during hydrogenation with and without catalysts has been investigated. Proceeding with the hydrogenation below 400° C. with molybdenum sulphide as catalyst, one preserves viscosity very well, obtaining viscous oils which are very stable. The viscosity increases in the higher fractions and the curves show more desirable material now than is present in the crude raw extract.

At temperatures of over 450° C. cracking predominates, obtaining principally low-boiling-point benzole-like hydrocarbons.

The crude raw stock has been described in Part I.[1]

The pressure vessel holding charges of 200 gms. of raw material consisted of a high-pressure shaking autoclave of V_2A steel, of 500 c. c. capacity, and sealed with special iron disks.[4]

The autoclave was placed in a special oven which is heated by gas. Temperatures were read with a nickel-copper thermo-element, inserted in a porcelain insulator-rod entering the cover of the bomb through a well in the center, and reaching into the reaction space of the bomb itself. One to two hours were necessary to reach reaction temperatures. Maximum temperatures were held constant two hours.

When we used the molybdenum catalyst we found it necessary to cool the apparatus after the first heating and vent the waste reaction gases and start up again after pumping in a fresh supply of hydrogen. An original charge of hydrogen at the start was found to be insufficient.

As source of hydrogen we employed electrolytic hydrogen. Reaction product was poured out of the vessel after finishing. The catalyst was removed by filtration. The percent methane and hydrogen was determined in the spent reaction gas after every run.

Using iodine as catalyst we would "activate" the autoclave; that is, in the case of naphthalene using 0.05 gm. iodine hydrogenation was continued until all the naphthalene had been completely converted in fluid products at 430° C., 100 atmospheres, and within two-hours reaction time. To accomplish this it was necessary to repeat the operation seven times; 0.05 gm. iodine were also used for the E-extract hydrogenations.

The molybdenum catalyst was prepared by evaporating to dryness a mixture of 5 gm. molybdic acid with 10 gm. silica-gel, and 25 c. c. distilled water.

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Analysis of Stresses In U-Bend Pipe Frame

JESSE YEAKEL, Structural Engineer

AS AN alternative to the solution of stresses due to expansion in U-bends in piping layouts by the use of charts such as given in piping catalogs and handbooks, the following method is offered.

This method is suggested by the column-analogy theory in the solution of indeterminate structures. To illustrate the method, an example is given below. The illustration is of a very simple form, but the theory itself could be applied to more complicated cases.

It is assumed that the dimensions of the bend, the pipe size, and the magnitude of the distortion, Δ, are known. The stresses and forces caused by the distortion are to be found. The steps to be taken:

(1) Locate the centroid of the frame. This locates the axis x — x.

(2) Determine the moment of inertia of the frame, $I_{x\text{-}x}$.

(3) Determine the magnitude of an imaginary force H in the formula

$$H = \frac{\Delta EI}{I_{x\text{-}x}},$$

where E is the modulus of elasticity of the material and I is the moment of inertia of the pipe section of which the frame is made.

(4) Calculate the moment at any desired point in the frame by means of the formula $M = H\bar{y}$, where M is the moment at the point whose distance from axis x — x is $\bar{y}$.

(5) Calculate the force at the anchor by statics.

(6) Calculate the flexural stress at any point in the frame by the formula $f_t = f_c = \frac{My}{I}$, where f_t = tensile unit stress, f_c = compressive unit stress, M = moment at the point in question, and $\frac{I}{y}$ = section modulus of the pipe.

Illustration: The assumed dimensions and pipe sizes are as shown in Figure 1. $\Delta = .9''$, which is about equivalent to the expansion for 700° F.

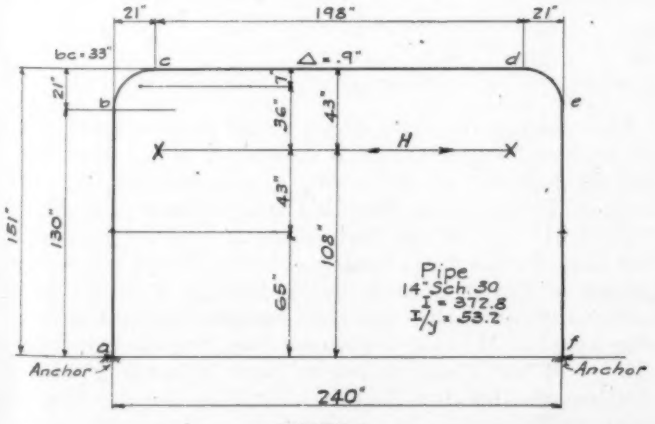


FIGURE 1

The necessary calculations follow, and are labeled with numbers given the six steps previously listed.

(1) Moments of the individual elements of the frame are taken about the base line. Since the cross-section of the pipe is uniform throughout the frame, the width of the pipe is taken as unity. The two rounded corners from a half-circle whose center of gravity is 14 inches above the horizontal diameter.

$2 \times 130 = 260$	$260 \times 65 = 16{,}900$
$2 \times 33 = 66$	$66 \times 144 = 9{,}504$
$1 \times 198 = 198$	$198 \times 151 = 29{,}898$
524	56,302

$\frac{56{,}302}{524} = 108''$, distance of x — x axis above base line.

(2) Distances from the x — x axis to the centroids of the elements of the frame can now be determined. They are shown in Figure 1. Calculations for $I_{x\text{-}x}$ should be made next, and are given below. No explanation seems necessary except to remind the reader that the moment of inertia of an element about the x—x axis is equal to $I + ah^2$, where I is the moment of inertia of the element about its own center of gravity, a is the area of the element, and h is the distance from the x — x axis to the center of gravity of the element. Also, the moment of inertia of the half-circle formed by the two corners about its own center of gravity is 2500. The frame is considered fixed at a and f.

$2 \times \frac{1}{12} \times 130^3$	=	366,000
$2 \times 130 \times 43^2$	=	480,700
half-circle	=	2,500
66×36^2	=	85,500
198×43^2	=	366,100
$I_{x\text{-}x}$	=	1,300,800

(3) $H = \frac{\Delta EI}{I_{x\text{-}x}} = \frac{.9 \times 30{,}000{,}000 \times 372.8}{1{,}300{,}800} = 7740\#$

(4) Moment at a, where $y = 108''$, is $M_a = 7740 \times 108 = 835{,}900''\#$.
Moment at b is $M_b = 7740 \times 22 = 170{,}300''\#$
Moment at c is $M_c = 7740 \times 43 = 332{,}800''\#$
Moments at d, e and f are the same as at c, b and a respectively.

(5) The force at a is

$$\frac{M_a + M_b}{130} = 7740\#.$$

The force at f is the same, but in the opposite direction. It will be noticed that this is the same as the value of H found in (3).

(6) Stress in pipe at $a = \frac{835{,}900}{53.2} = 15{,}700$ psi.

Stress in pipe at $b = \frac{170{,}300}{53.2} = 3{,}200$ psi.

Stress in pipe at $c = \frac{332{,}800}{53.2} = 6{,}300$ psi.

It should be stated that the stresses and forces determined here are for the effect of the expansion only, and not for any loads due to weight of the pipe or the flow of materials within the pipe. Also, the frame is restrained against turning at points a and f, but not at any point between.

The method outlined here can be applied to a bend of any shape so long as there is an axis of symmetry at right angles to the direction of the distortion. It can be used for any elastic material so long as the distortion is within the elastic limit of the material. If there is no axis of symmetry—for instance if the side legs were unequal in length—a solution could also be found by the column analogy theory, and the procedure would be only slightly more complicated than shown here.

It will be noted that this method automatically takes into account the effect of the rounded corners. It is not necessary, as in some methods, to calculate for square corners and then make an adjustment for the rounding of the corners. Also much greater accuracy can be attained than is possible with the use of charts. This is of minor importance in the case of small pipe sizes, but in some cases where the pipe section is heavy and distances are relatively great, more accuracy may be desirable.

Physical Chemistry of Lubricating Oils

V. THE STATISTICAL ANALYSIS OF LUBRICATING OILS

A. BONDI

DURING the past fifteen years considerable progress has been made in the elucidation of the composition of lubricating oils. The realiaztion that separation of lubricating oils into individual compounds, or even the isolation out of same of well-defined chemical substances in the conventional manner of organic chemistry was entirely hopeless, and had to be replaced by fundamentally new methods of attack was the first step in the direction which has since proven fruitful. The new method, introduction of which is difficult to trace, embodies two separate lines of attack: (a) The determination of the statistical distribution of molecule sizes and chemical-bond types in the oil fraction under consideration, and (b) The synthesis of hydrocarbons similar in chemical constitution to the "average" determined by methods (a) and comparison of their physical properties with those of the oil fraction. The narrower the molecule size and type range of the recovered lube fraction, the more likely it is that its constituents resemble the model hydrocarbon in chemical structure. Various steps of this physico-chemical method of analysis will be outlined in the following paragraphs.

V. 1 Molecule Size Distribution in Lubricating Oil Fraction (Distillation Analysis)

The average molecular weight of lubricating oils has, during the past few years, developed out of a curiosity into a significant physical constant. This is mainly due to the fact that the various methods of analysis for "chemical-bond distribution," which will be discussed later on, require a knowledge of the average molecular weight. The standard methods of determination (either by ebulliometry or cryoscopy) have been investigated by a number of authors[143-147] as to their suitability for lubricating-oil analysis. They found,[143, 147] and this is a bit disconcerting, various types of lubricating oils to interact with the solvent used in a somewhat unpredictable manner. They advise, therefore, either to make the determination at more than one concentration and extrapolate to zero concentration (and even then there will be discrepancies between cryoscopic and ebulliometric measurements) or to standardize on just one method to obtain self-consistent results. All of these methods are based on the validity of Raoult's law for the solution of lubricating oil in the solvent, which gives the molecular weight M by

$$M = \frac{RT^2}{\Delta H} \cdot \frac{c_L}{1000 c_S \Delta T} \qquad (1)$$

where T = melting (or boiling) point in ° K, ΔH = latent heat of fusion (or vaporization) of solvent, ΔT = melting point depression (or boiling point increase) in ° C, c_L = gram of lubricating oil, c_S = grams of solvent.

The technique and appropriate precautions which have to be observed in the actual determination are discussed in the references given and in most textbooks on physico-chemical methods of analysis. Attention is here called to the nature of the "molecular weight" so determined. The use of Raoult's law in the derivation of Equation (1) implies that we actually do not determine the molecular weight, but the number of molecules per unit weight, or better, the weight of a certain number, N, of molecules ($N = 6.02 \times 10^{23}$). For pure substances the weight of N molecules is equal to the "chemical" molecular weight, and $M/N = m$ = mass per molecule. Such reasoning does not apply to mixtures. The "average molecular weight" M^N of a mixture, such as lubricating oil, is not the arithmetical average of the molecular weights of all components but is given by

$$\frac{100}{M^N} = \frac{P_1}{M_1} + \frac{P_2}{M_2} + \frac{P_3}{M_3} + \ldots\ldots\ldots\ldots (2)$$

where P_i = weight percentage of each component.

This means that the mean molecular weight of a mixture of 50 percent of a substance with M = 250 and 50 percent of M = 500, if determined by any method derived from Raoult's law will **not** give M = 375 but M^N = 330! In high polymer chemistry where one also always deals with mixtures, M^N is generally known as "number-average molecular weight." For pure compounds this and the "weight-average molecular weight" M^W are of course alike, but for mixtures $(M^W - M^N) > O$ and increases with increasing width of molecule size distribution. A 1:1 mixture of bright stock cylinder oil (M = 800) and neutral oil (M = 300) would give M^N = 440 and M^W = 550. Comparison with the example given above shows the rapid divergence of the two average molecular weights with "inhomogeneity" of the blend. As many other physical properties of mixtures, such as vapor pressure and viscosity, very often are dominated by

the property of the low-molecular-weight component, the dominating influence of the low-molecular-weight components on the number-average molecular weight is not of any serious consequence. For the proper interpretation of data, however, it is well to keep in mind that nearly all average molecular-weight data of lubricating oils in the literature are derived by application of Raoult's law and are therefore "number-average molecular weights." M^W could be determined by measuring the sedimentation rate in an ultra centrifuge, the scattering of light, or the viscosity of dilute solutions. The sedimentation rate of lubricating oils out of solutions has not yet been reported, although the separability of bright stocks out of petroleum-ether solutions by means of ultra centrifuge has been observed. The light-scattering method is not very promising for such strongly fluorescing materials (of relatively low M), as lube oils. The molecular weight determination by viscometry of dilute solutions of lubricating oils in toluene has been proposed by Tausz,[148] but mathematical analysis of his data by the reviewer proved that the specific effects of molecular interaction between solvent and solute on viscosity are too strong to make this method appear reliable.

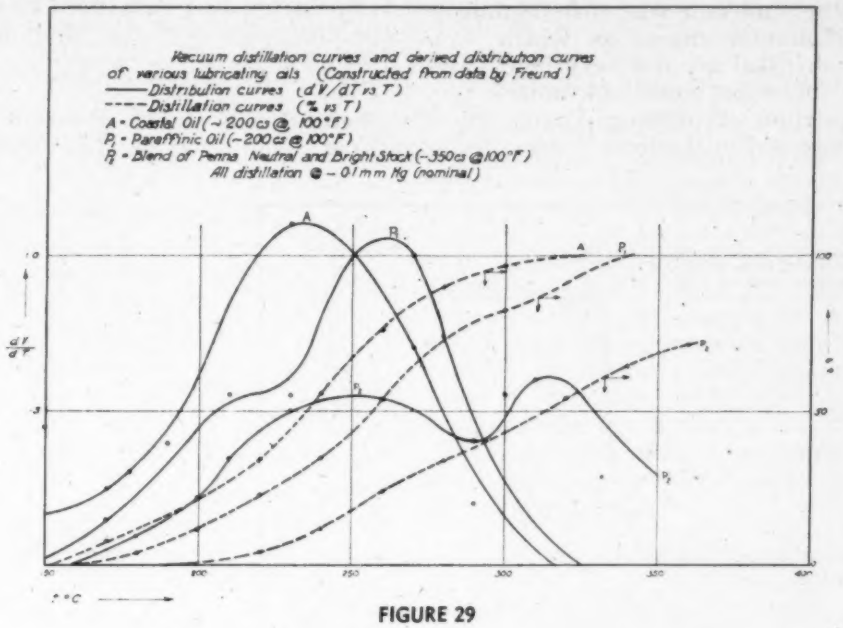

Vacuum distillation curves and derived distribution curves
of various lubricating oils (Constructed from data by Freund)
——Distribution curves (dV/dT vs T)
----Distillation curves (% vs T)
A = Coastal Oil (~ 200 cs @ 100°F)
P1 = Paraffinic Oil (~ 200 cs @ 100°F)
P2 = Blend of Penna Neutral and Bright Stock (~ 350 cs @ 100°F)
All distillation @ ~ 0.1 mm Hg (nominal)
A
B
P1
P2
°C

FIGURE 29

TABLE 38

Boiling Points of Various Hydrocarbons as Function of Their Molecular Weight.

Hydrocarbons	B.P. @ 1 mm. of Maximum Number of Compounds	Lowest Observed	Highest Observed In Group	
C_{24}	195 °C. ±5 °C.	181	210.5	..
C_{25}	202 °C. ±5 °C.	180	212	..
C_{26}	207 °C. ±5 °C.	...	...	..
C_{28}	212 °C. ±5 °C.	209	220	..
C_{30}	222 °C. ±5 °C.	...	...	..
C_{36}	231 °C. ±5 °C.	...	242	..
C_{43}	235 °C.	...	...	..
C_{45}	250 °C.	...	260	..
C_{48}	308 °C.	...	...	..

TABLE 39

Vapor Pressure and Latent Heat of Varporization of Various Hydrocarbons as Function of Their Molecular structure.[164]

	B.P. @ 1 mm. Pressure	ΔH_{vap} (Cal/mole)	Δh_{vap} (Cal./G)
3—EL-5-(2-Et-But)-Octadecane...	180 °C.	20,900	57
11—Neo pentyl heneicosane.....	189 °C.	22,000	60
11—Cyclo pentyl heneicosane.....	198 °C.	21,850	60
5—Cyclo hexyl eicosane.....	202 °C.	23,300	64
2—Cyclo hexyl eicosane.....	207 °C.	23,700	65
1—Cyclo hexyl eicosane.....	212 °C.	22,600	62
1—Phenyl eicosane.....	205 °C.	22,600	63
1.1—Diphenyl tetradecane.....	207 °C.	23,400	66.7
n—Hexacosane.....	205 °C.	22,400	61

The next question is for the molecule-size distribution in the lubricating oil sample at hand. Of the many fractionating methods which are available, hardly any can be considered as separating lube oil components by molecular weight only. A more or less pronounced effect of "group" separation is always superimposed upon the fractionation results. Molecular (short path) distillation comes closest to the ideal, since here the "distillability"[149] depends on the ratio $P/\sqrt{M}$, p = vapor pressure of component and M its molecular weight. Due to the nature of the operation the achievable degree of separation is very low, however. The next best method is ordinary vacuum distillation, the effectiveness of which is based on the vapor-pressure ratios of the components. It so happens that the vapor pressure of the higher hydrocarbons depends more on the molecular weight than on chemical constitution (Table 38); distillation curves provide, therefore, a fairly good picture of the mole-size distribution. All other methods follow in the order (steam vaporization, gas-precipitation, adsorption, extraction) as to their ability to resolve a hydrocarbon mixture according to the molecular weight of its components. As much as the boiling point of higher-molecular-weight hydrocarbons is conditioned by the molecular weight, one should always remember that it is still to some extent affected by the chemical constitution. The data on Table 39 bring this out quite clearly. Another, but not quite as important a point, is the difference in ΔH_V, the heat of evaporation, between the various types of hydrocarbons, the effect of which is, that the properties and composition of the distillation fractions of a lubricating oil may vary somewhat, depending upon the absolute pressure at which the separation had been performed. As a rule it will be found that boiling points and ΔH_V increase in the

order paraffinic < naphthenic < aromatic < condensed ring aromatic and naphthenic hydrocarbons.

Being fully conscious of the limitations thus imposed upon the evaluation of such data, we proceed to the inspection of vacuum-distillation curves of lubricating oils. These are the most readily available representation of the molecule-size distribution in oils. The usual distillation charts on which T is plotted vs. percent distilled are not very instructive when there are no "plateaus," and look indeed very much alike. The method of plotting dV/dT vs. T which has been suggested by Lederer[150] provides us on Figure 29. Sometimes the temperatures of distillation or "boiling points" of the various fractions have not been given, then the molecular weight can be employed as independent variable and a distribution function dV/dM vs. M be constructed. If instead of vacuum distillation, steam distillation at uniform temperature had been used to obtain an analysis of the composition of the fraction at hand, the distribution curve can be obtained by plotting $\frac{dV}{dR}$ vs. R, where R = Ratio of water to oil weight distilled. From some such data[151] Figure 31 has been constructed. If the vapor pressure of the fraction had also been measured, the molecular weight could be calculated readily from R by Dalton's law.

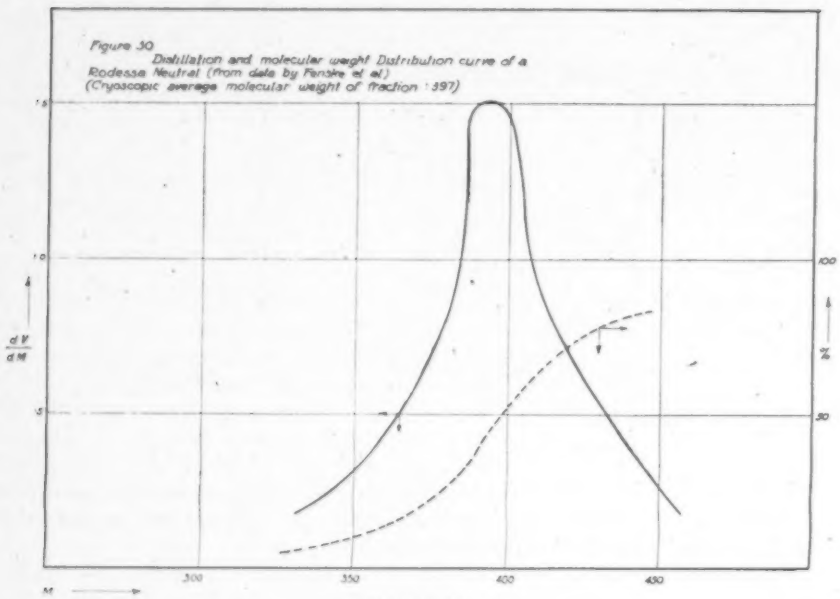


FIGURE 30

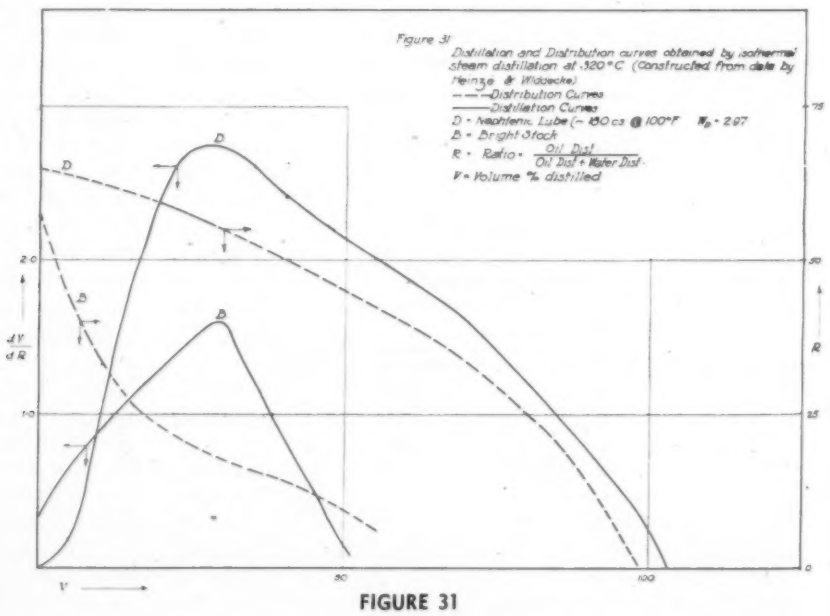


Figure 31
Distillation and Distribution curves obtained by isothermal steam distillation at 320°C (Constructed from data by Heinze & Wildsecke)
——Distribution Curves
———Distillation Curves
D = Naphtenic Lube (~ 150 cs @ 100°F
B = Bright Stock
R = Ratio = Oil Dist / Oil Dist + Water Dist
V = Volume % distilled
D
B
dV/dR
R
V
50
100

FIGURE 31

For the technical evaluation of lubricating oils, there is little need of distillation analysis, any one of the many evaporation tests usually giving sufficient information about their volatility. Only if information about the presence of residual oils is desired or refining procedures be prepared, does vacuum or steam distillation find application in the control laboratory. Correlation between distillation results and with a much better picture of the sought distribution function, as is evidenced from Figures 29 and 30. Those graphs do not only permit an interesting control of the efficiency of fractionation procedures used by the refiner but also indicate whether the sample at hand is a blend or a single fraction. If an oil had been blended up out of fractions differing widely in boiling range, as is common practice in lube-oil manufacturing, one can estimate from the dV/dT vs. T graph the percentage of "low"- and "high"-boiling material employed by the supplier, as is indicated

carbon-forming tendency of lubricating oils as was once proposed[152] has proven of little value. We know now[90] that carbon formation is essentially a function of the concentration of non-hydrocarbons and highly condensed cyclic hydrocarbons which can be separated as "petroleum-resins" by selective adsorption.

For the investigation of lube-oil composition it is, however, important to obtain a clear picture of the molecule-size distribution in the sample at hand (if it is not possible to produce—as is most desirable—very "narrow" cut fractions) for the proper interpretation of the other physical data. As we have seen, distillation curves can provide us with such picture.

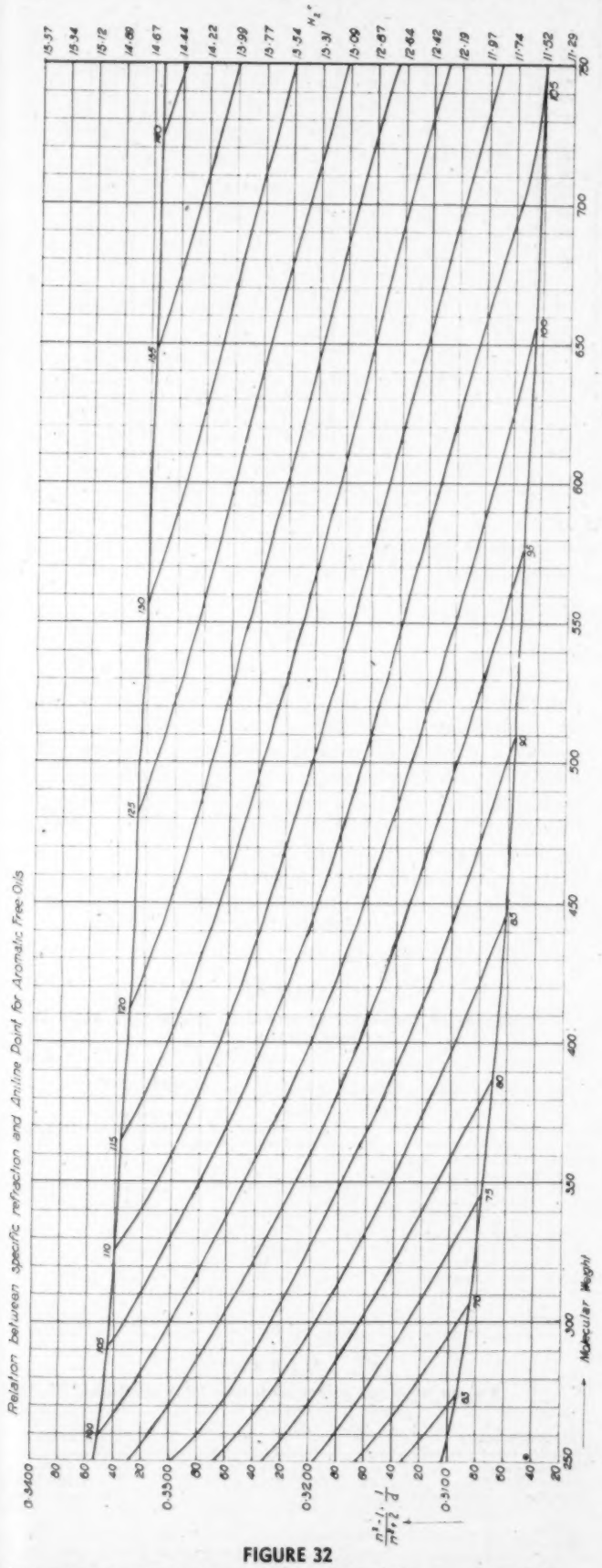


FIGURE 32
Waterman-Graph relating Aniline point-specification refractivity-average Molecular weight of saturated hydrocarbons.

V. 2 Hydrocarbon Type Distribution

Following separation of a lubricating oil into a number of fairly "homogeneous" fractions with respect to molecular-weight distribution there are two ways open for the separation of these fractions into portions enriched in certain hydrocarbon types. One way is selective adsorption, the other selective extraction. As we shall see in Section VI, both methods tend to separate aromatic from completely saturated compounds. Adsorption is especially suited to the removal of aromatic and non-hydrocarbons from all other compounds, while extraction with selective solvents permits subsequent subdivision into naphthenes and paraffinic substances. At this point we shall digress for a while to discuss the presently available schemes for the

V. 3 Chemical Bond Distribution Analysis in Lubricating Oils

The recognition of the impossibility to isolate individual chemical compounds from lubricating oils, regardless how close the boiling range and how far the degree of uniformity of composition could be driven, brought to the forefront those methods of analysis which would indicate the relative distribution of aromatic, naphthenic, and paraffinic compounds in hydrocarbon mixtures. Chemical methods, such as successive sulfonation of olefines and aromatics, which had proven quite successful in gasoline analysis, were also applied to lubricating oils[153] usually in connection with measurement of refractivity and aniline points—but did not always yield consistent results. The difficulty of selecting a sulfuric acid treatment which was sufficiently drastic to remove all aromatic compounds and at the same time not to attack any of the saturated compounds became nearly unsurmountable. The fact that a number of lubricating oils, such as certain Pennsylvania stocks and many bright stock blends cannot at all be freed quantitatively from aromatics by acid treatment and the akwardness of the entire procedure led to the complete abandonment of chemical methods in lubricating-oil analysis. Recourse has instead been taken to the evaluation of physical properties with the help of statistical methods. The first complete analysis of this type was proposed by Waterman and his associates.[154] It enjoys still today a large measure of popularity in spite of a number of serious shortcomings. For the calculation of percent carbon in aromatic, naphthenic and paraffinic bonding, there are required: specific gravity d_4^{20}, refractive index n_D^{20}, average molecular weight M^N, aniline point °C (before and after hydrogenation of oil). The method is based on the relationship between specific refraction [r], which was mentioned in Section IV. 3 and chemical bonds of saturated hydrocarbons. The change (increase) in aniline points by hydrogenation is a measure of the percentage of carbon atoms in aromatic rings. After having made a large number of determinations, Waterman found that the hydrogenation could—at the expense of some accuracy—

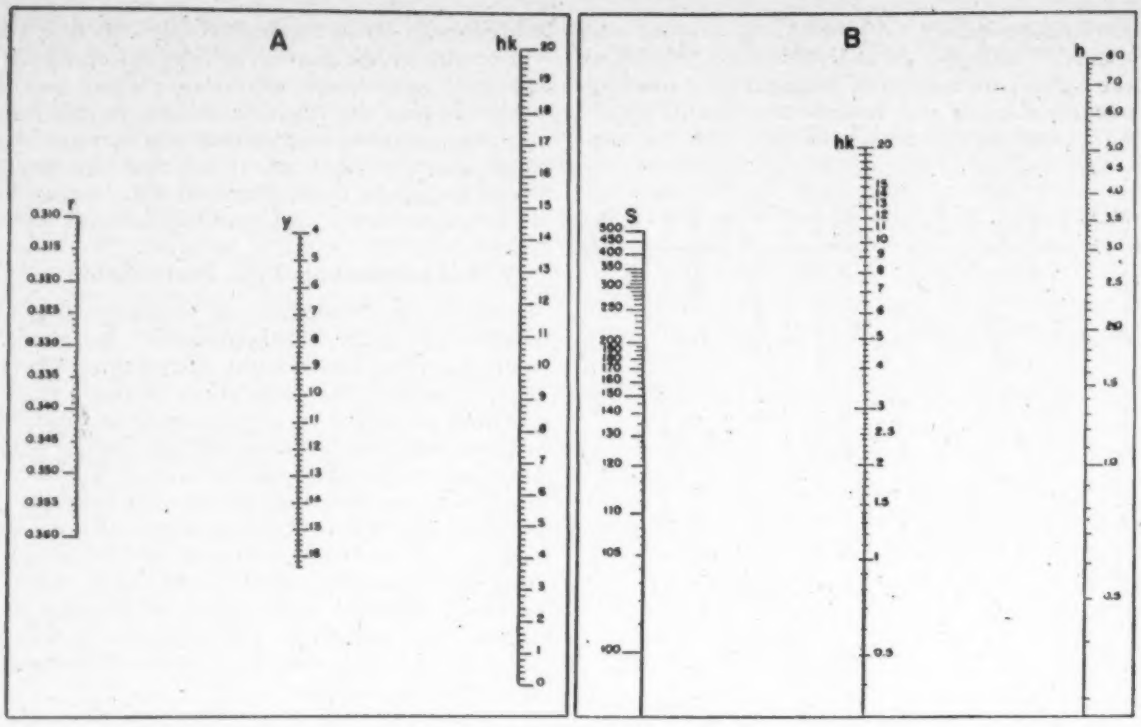


FIGURE 33

Nomographic charts for solution of general equations.

$$A,\ hk = 201.6r - 1.6978y - 42.02$$

$$B,\ h = \left[\frac{hk}{0.811\ (S-98)^{1.412}}\right]$$

(From Deanesley and Carleton.[157])

be dispensed with, and the procedure is now as follows:

Determine the hypothetical aniline point P_i corresponding to the $[r]_{obs}$ and the M^N of the sample from Figure 32. Multiply the difference between this hypothetical aniline point and the observed point with 0.8:

$$(P_i - P_{obs}) \times 0.8 = \Delta P$$

Add ΔP to the observed experimental aniline point P_{obs} of the sample:

$$P_{obs} + \Delta P = P_h$$

P_h is the aniline point which the oil would have, had it been hydrogenated. Now read from the graph the specific refractivity $[r]_h$ corresponding to P_h and M of the sample. Also read the specific refractivity $[r]_s$ of the fully saturated hydrocarbons (highest line) and the naphthenes of the lowest line for the molecular weight M. We obtain:

$$\%C \text{ in naphthene rings: } 100 \times \frac{[r]_s - [r]_h}{[r]_s - [r]_N}$$

$$\%C \text{ in aromatic rings: } 100 \times 0.85 \times 0.80 \times \Delta P$$

%C in paraffinic side chains:

100 — (% Aromatic + % Naphthenic)

The main source of inconsistencies, which various investigators[155,156] observed in the application of Waterman's analysis to substances of known composition, seems to be the use of the aniline point besides of the discrepancies in his refractivity increments from the "true" values.* Table 40 shows that the aniline point is not only a function of the relative amounts of naphthenic and aromatic rings in a molecule but is also greatly influenced by its shape. The solubilizing influence of highly aromatic compounds on saturated hydrocarbons is an additional (most serious) source of interference. Elimination of any solubility measurements from hydrocarbon analysis would therefore be most desirable. Deanesley and Carleton[157] accomplished this by using a statistical (empirical) relationship between the dispersivity (Gladstone-Dale) and the refractivity increment per double bond as measure of the aromatic unsaturation. They also use (a slightly improved version of) the relation between [r] and the hydrogen content of

TABLE 40

Influence of Molecular Structure on the Aniline Point of Hydrocarbons.[161]

	ANILINE POINT		
	Observed	Waterman's Graph	Difference
13—Methyl Pentacosane $C_{26}H_{54}$	116.0 °C.*	115.6	+0.4
5—n Butyl docosane $C_{26}H_{54}$	114.0 °C.	115.6	—1.6
6,11—di n Amyl hexadecane $C_{26}H_{54}$	112.0 °C.	115.6	—3.6
5,14—d n Butyl octadecane $C_{26}H_{54}$	111.3 °C.	115.6	—4.3
1—Cyclo hexyl eicosane $C_{26}H_{52}$	110.8	111.0	—0.2
3—Cyclo hexyl eicosane $C_{26}H_{52}$	107.7	111.0	—3.3
9—Cyclo hexyl eicosane $C_{26}H_{52}$	106.4	111.0	—4.6
2—Phenyl eicosane $C_{26}H_{46}$	57.6	.....	.....
4—Phenyl eicosane $C_{26}H_{46}$	61.0	.....	.....
9—Phenyl eicosane $C_{26}H_{46}$	63.4	.....	.....

* From W. M. Mazee. T.O.M. Reel No. 79 p. 2504-77.

TABLE 41

Values of k for Representative Hydrocarbons.[157]

Hydrocarbon	k at 20 °C.
Cyclo pentene	1.46
Alkyl cyclohexane	1.40—1.86
Aliphatic mono olefine	160—1.90
Alkyl benzenes	160—1.90
Indene	1.89
Diphenyl	1.91
Alkyl naphthalenes	2.10—2.25
Alkyl anthracenes	2.60—2.90

* It would, indeed, be wise to remember that bond-increments of any physical property are definitely limited as to the accuracy of their numerical values since the underlying assumption of their freedom from vicinal effects is, at best, a first approximation only.

TABLE 42

Comparison of the Accuracy of the Statistical Methods of Hydrocarbon Analysis by Waterman and Coll., and by Deanesley and Carleton Against Pure Hydrocarbons of Known Composition.

Hydrocarbon	Type of C-Bond	Theory	Waterman I	Waterman II*	D-C†	L.A.‡
6,11-di-n Amyl-hexadecane	Paraffinic	100%	92.7	92.7	99.57	88
	Naphthenic	0%	5.6	6.4	.43	12
	Aromatic	0%	1.7	.9	....	....
11—Cyclohexyl-heneicosane	Paraffinic	77.8	77.4	77.4	73.4	80
	Naphthenic	22.2	22.6	22.6	26.6	20
	Aromatic	0	0	0	0	0
11—Phenyl heneicosane	Paraffinic	77.8	71	71	70.5	....
	Naphthenic	0	—3	6.8	—1.3	....
	Aromatic	22.2	32	22.2	30.8	....
11—Decalir-heneicosane	Paraffinic	67.7	67.8	67.8	65	64
	Naphthenic	32.3	30.4	31.0	35	36
	Aromatic	0	1.8	1.2	....	....
1—B-Stearyl-Tetralin	Paraffinic	64.2	54.5	54.5	83	....
	Naphthenic	14.3	8.2	19.7	—14.3	....
	Aromatic	21.4	37.3	25.8	31.3	....
1—Butyl-Stearyl-1-B-Naphthalin	Paraffinic	68.8	77.8	77.4	82.5	....
	Naphthenic	....	—29.1	—13.4	—17.5	....
	Aromatic	31.2	51.7	36	37.8	....

* Using factor 0.47 to convert Aniline point difference to percent aromatic C-atoms, as proposed by Schiessler et al (164), which seems to give indeed better results throughout.

† Deanesley-Carleton Method (157).

‡ Lipkin and Kurtz's Naphthene Ring Analysis of saturated hydrocarbons from density-temperature coefficient vs. density relation (8).

In order to show how far the statistical methods of analysis may occasionally deviate from reality, some of the most unfavorable cases have been assembled in this table.

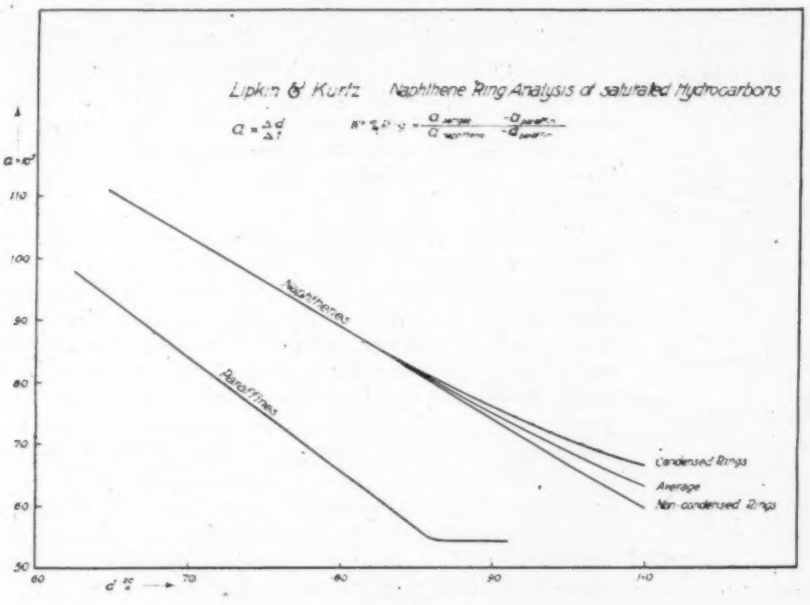


TABLE 43

Unit Coefficient of Molar Extinction Coefficients in Near Infrared of Various Hydrocarbons.[139]

Wave Number cm⁻¹	Group Sought	—CH₃ α′	>CH₂ β	→CH γ	>CH Aromatic δ	Av. Deviation in Groups per Molecule in n Paraff.	iso Paraff.	Naphth.	Arom.	arom. h.*
5668	>CH₂	0.0529	0.0638	.....	0.0216	....	0.5	0.8!	0.5	1.8
5781	>CH₂	0.0708	0.0908	.....	0.0137	....	0.5	+1.2!	0.6	0.6
5800	—CH₃	0.1052	0.0674	.....	0.0138	....	0.6	+1.1!	0.5	+1.8!
5860	—CH₃	0.1363	0.0194	.....	0.0314	....	0.3	0.3	0.4	0.7
5870	—CH₃	0.1426	0.0130	.....	0.0376	....	....	....	....	....
5900	—CH₃	0.1256	0.0057	.....	0.0764	....	0.4	0.3	0.4	0.7
	>CH (ar)						0.6	0.6	0.6	1.7
6850	→CH	0.00718	0.00748	0.00532	0.00175	....	1.0	—1.2	0.5	—2.8!
6973	—CH₃	0.00948	0.00776	.....	0.00136	....	0.4	0.4	0.4	—0.6
7064	>CH₂	0.01253	0.00956	.....	0.00134	....	0.7	—0.9	0.2	1.1!
7185	>CH₂	0.01791	0.00978	.....	0.00118	....	0.4	+0.2	0.4	1.3!
7350	—CH₃	0.01025	0.00164	.....	0.00184	....	0.3	0.3	0.3	—1.5
8160	→CH	0.00197	0.00992	0.01132	.....	0.5	0.5	0.2	0.2	0.8
8254	>CH₂	0.00629	0.01989	.....	.....	0.2	0.5	—0.3	0.2	1.3
8300	>CH₂	0.00948	0.01585	.....	.....	0.2	0.8	+1.4!	0.3	0.8
8400	—CH₃	0.02705	0.00735	.....	.....	0.1	0.3	+0.6	0.3	+0.5
8680	—CH₃	0.00663	0.00176	.....	0.00622	0.2	0.3	—0.3	1.3!	1.4!
8750	>CH (ar)	0.00327	0.00126	.....	0.001121	0.1	0.2	—0.3	0.6	1.4!

Molar extinction coefficient $K = n_a\alpha' + n_b\beta + n_c\gamma + n_d\delta$, where n_a, n_b, n_c, n_d are the number of —CH₃, >CH₂, →CH, and >CH aromatic groups per molecule respectively.

* Av. deviations in high molecular weight aromatic hydrocarbons (C_{24}—C_{34}). The large deviations, if considered percentage-wise, still represent a fair degree of accuracy.

saturated hydrocarbons. Required data are: specific gravity $d\frac{20}{4}$, refractive index $n\frac{20}{D}$, dispersion $\frac{n_F - n_C}{d} \times 10^4$, Bromine N. (for samples containing olefines), average molecular weight M, ultimate analysis = wt. % H, wt. % C.

The procedure is as follows: calculate $[r]_D$ (Lorehtz Lorenz), $Y = 100\ \frac{\%H}{\%C + \%H}$, $S = \frac{n_F - n_C}{d} \times 10^4$, then determine by means of nomograph 33 the constants hk and h, calculate from Bromine Number (if any)

$$h_o = \frac{Br - No.}{79.3}, \text{ then we get: } h_A = h - h_o$$

$$\% \text{ aromaticity } A = \frac{1191.5 \times h_A}{100 - Y}$$

$$\text{C-atoms per molecule } n = \frac{M\,(100 - Y)}{1201}$$

$$\text{Rings per molecule } R = 1 - \frac{M \times h}{201.6} + \frac{M\,(2.016 - 0.14026Y)}{24.212}$$

$$\text{Aromatic C-atoms per molecule } C_A = \frac{nA}{100}$$

From Table 41 one obtains the dominating kind of aromatic ring, where $K = \frac{hk}{h}$. This leads to a reasonable assumption of the average number of carbon atoms C_R per ring giving:

Number of ring carbon atoms per molecule = $R \times C_R$

Naphthenic C-atoms per molecule = $C_N = R \times C_R - C_A$

Alkyl C-atoms per molecule = $C_P = n - (C_A - C_N)$

The percentage each of aromatic, naphthenic and alkyl carbon atoms is then obtained in an obvious manner. This method has proven more consistent but somewhat less reliable than Waterman's; due to the requirement of rather accurate ultimate analysis it has not acquired much popularity. The assumptions made are:

(a) that all values of k fall on (or close to) the curve:

$$k = 0.811\left(\frac{S - 98}{h}\right)^{0.298}$$

which seems to be the case most of the time.

(b) that k is additive with respect to the type of aromaticity (or unsaturation) involved.

For the majority of lubricating oils, the requirement of a bromine number determination can safely be dropped since olefinic unsaturation does not normally occur in same. The reviewer has always found Br and I number of lubricating oils to be due to substitution rather than addition, as was brought out by subsequent determination of the liberated hydrogen halide.

In order to examine the degree of accuracy which could be expected from the Waterman and Deanesley - Carlton methods, the appropriate calculations were carried out for the pure hydrocarbons described by Cosby and Sutherland[6] and the data assem-

TABLE 44

Comparison of the Properties of Closely Cut "Constant-Boiling" Fractions* of a Mid-Continent Petroleum With Those of Pure Hydrocarbons of Known Composition.

SUBSTANCE	n	x	Percent Par.	R‡	Percent Naph.	100 °F. Stokes	210 Stokes	A-P† °C.
Fraction A-6	27.6	—2.8	67	1.5 (1.8)	33	0.489	0.0606	104.1
1 Cyclohexyl-2-cyclohexyl methyl) pentadecane	28	—2.0	57	2.0	43	0.403	0.506	105.1
Fraction A-Res. 11—	28	‡0.35	79	1.0	21	0.181	0.0398	113.3
Cyclohexyl heneicosane	27	0.0	78	1.0	22	0.175	0.0375	108.5
Fraction C-15	29.6	—3.0	58	2.1	42	0.589	0.071	106.5
5,14-Dicyclohexyl Octadecane	30	—2.0	60	2.0	40	0.513	0.064	....
Fraction D-30	32.2	—1.31	73	(1.72)	27	0.405	0.0634	115
11- -Decatin heneicosane	31	—2.0	68	(2.0)	32	0.457	0.0636	108.9

* Fractions are from Acetone Extraction of "Water-White" Oil, B. J. Mair and S. T. Schicktanz, Ind. Eng. Chem. 28 · 1446 (1936).

† Aniline Points of Mair's saturated fractions are higher throughout than Waterman's diagram "permits", a phenomenon which has not—or at least not to that extent—been observed with pure synthetic hydrocarbons.

‡ Rings per molecule.

bled in Table 42. The Deanesley-Carleton analysis appears on the whole unreliable and gives especially erratic results for condensed ring naphthenes.

It seems worth while to compare the percentage naphthenes as calculated from the expansion coefficient method of Kurtz[8], which was reported on in Section I. 2, with the results of the Waterman and the Deanesley-Carleton analyses.

A hydrocarbon type analysis for lubricating oils based on density, refractivity and ultimate analysis has also been proposed by Umstatter,[158] but it seems to be incomplete; at least the reviewer was not able to obtain satisfactory results with this method.

There is doubtless room for improvement in the field of statistical hydrocarbon analysis, but it is difficult right now to see what readily determined physical property will simplify or improve on the existing procedures. Constants which depend on the surface tension such as the Parachor or the total surface energy for both of which bond increments have been successfully applied in organic chemistry, are ruled out, since we know now that the surface of lubricating oils differs in composition from the bulk of the liquid. A significant advance has recently been made by a number of workers of the United States Bureau of Standards.[159] Their method is based on the observation that the characteristic frequency of vibration of the carbon-hydrogen bond in the near infrared is strongly dependent on the nature of the neighboring carbon-carbon bond. Primary, secondary, tertiary, and aromatic carbon bonds can be well differentiated in this part of the infrared spectrum (5400 to 8900 cm^{-1}). The extinction coefficient at the respective characteristic frequencies is then a measure of the (relative) abundance of each type of carbon bond in the sample. The preliminary investigations showed that a fair degree of accuracy can be obtained by this method of bond analysis (Table 43). The present availability of a large number of pure synthetic hydrocarbons for calibration purposes and of many infrared spectroscopes in the petroleum industry may inaugurate considerable activity in the application of this new method to hydrocarbon type analysis of petroleum fractions.

Waterman analyses of a number of lubricating oil fractions, together with some other data have been assembled on Figure 34. All of these data have been obtained after fractionation by vacuum distillation. One can see that the relative abundance of aromatic, naphthenic and paraffinic carbon bonds changes but little from one distillation fraction of an oil to the next (which means that the number of rings per molecule, as a rule, increases steadily with the boiling point of the fraction.) Solvent extraction, the effects of which have been presented on Figure 35, separates visibly by hydrocarbon type. There are insufficient data available to permit definite statements as to the ability of certain solvents to "discern" between aromatic and condensed ring cyclo-aliphatic hydrocarbons. From presently available evidence, it appears to the writer that the degree of separation between groups of hydrocarbons which solvent extraction does or does not achieve is not as much a function of the solvent as of properly conducting the extraction, as had first been emphasized by Varteressian and Fenske[160] and Saal and v. Wijk.[161] Complete or nearly complete resolution of a lubricating-oil fraction into hydrocarbon-type groups seems to be achievable only by combination of repeated distillation, adsorption and extraction steps such as was performed by Rossini[162] and numerous subsequent investigators. As a rule the best procedure is to perform first a rough vacuum fractionation of the long residuum (or lube fraction on hand) followed by two or more redistillations of the cuts obtained, each time combining those cuts of equal physical properties[163] then subjecting the cuts to a "rough" extraction with a very selective solvent, refining the raffinate with a non-activated adsorbent, redistilling

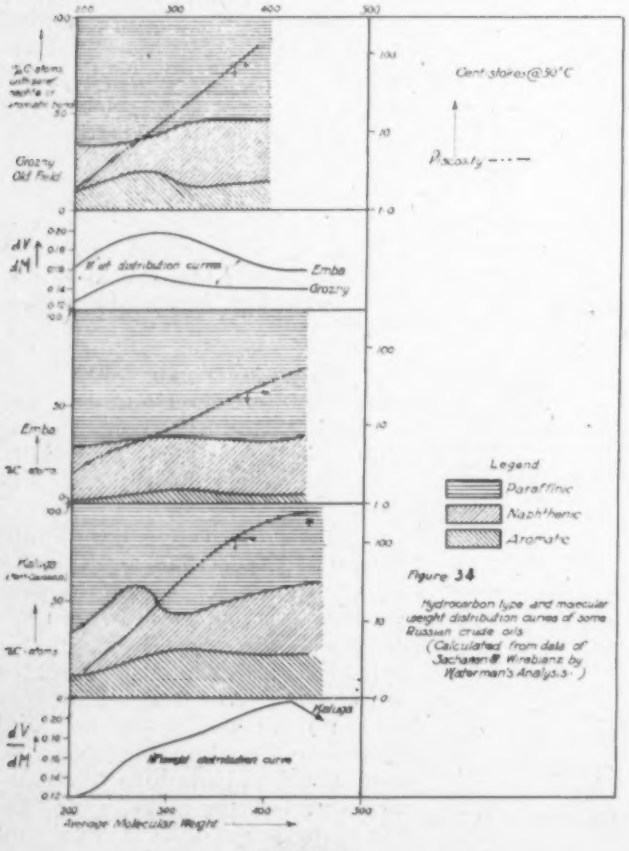


Figure 34

Hydrocarbon type and molecular weight distribution curves of some Russian crude oils (Calculated from data of Sachanen & Wirabianz by Waterman's Analysis.)

FIGURE 34

the raffinate each into "constant-boiling" fractions followed by careful reflux-extraction. The fractions thus obtained—being in the case of Rossini's work about 1/40,000 of the crude petroleum each—are about the closest one has so far come in the isolation of "pure" hydrocarbons from lubricating oil. There can, however, be no question but that each of these narrow fractions is still a mixture of several probably unresolvable isomers.

cyclic extract portion of lubricating oils is by far not as readily understood. Especially the extreme drop of viscosity on hydrogenation of the extract portion is something that could so far not be duplicated with synthetic compounds and has probably to wait for an explanation until more synthetic hydrocarbons of highly cyclic structure have been produced and/or until we have gained a clearer understanding of the nature of the not inconsiderable non-hydrocarbon

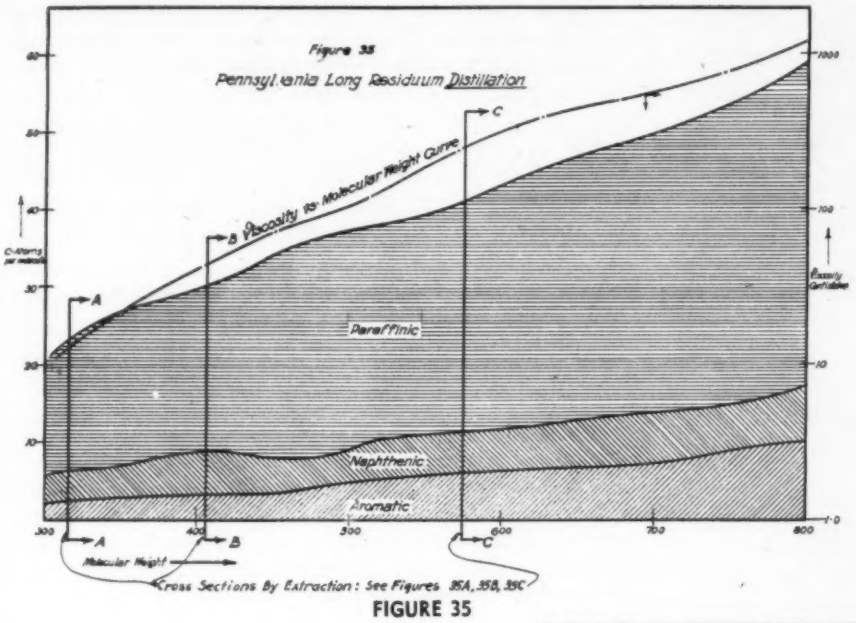
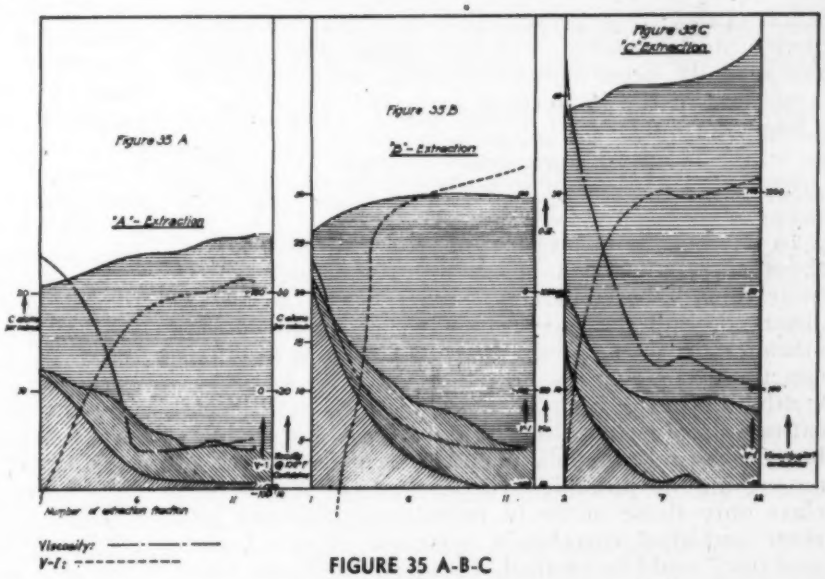


FIGURE 35



Graphs constructed from data by Kapff, Bowman, and Lowy.[168]

FIGURE 35 A-B-C

V. 4 The Probable Structure of the Hydrocarbons in Lubricating Oils.

The narrow fractions obtained by the method outlined above are sufficiently "homogeneous" in chemical composition to permit comparison of their physical properties and carbon-bond distribution with those of synthetic pure hydrocarbons of known structure. Ample data are now available[164, 165] on the physical constants of pure hydrocarbons. The tabulated comparisons between the closely-cut lubricating-oil fractions and the pure substances on Table 44 are very gratifying and show that the statistical methods of analysis do, indeed, convey a fair picture of the chemical structure of the predominant hydrocarbons in lubricating oils, at least as far as the more saturated fractions are concerned. The structure of the highly

portion of the solvent extracts. Judging by their sulphur and oxygen content, some solvent extracts seem to contain up to 30 or 40 percent non-hydrocarbons unless these compounds contain excessively large numbers of sulphur and oxygen atoms per molecule. If the latter is the case, it is conceivable that some potentially valuable organic chemicals are, together with the extract, presently going to fuel! Elucidation of the chemical structure of these compounds may therefore pay dividends, not only in additional knowledge.

A number of authors[166-69] have been able to correlate the various physical properties of petroleum fractions as well as its average chemical composition (C:H Ratio) in a unified manner, mainly on the basis of the specific gravity. The best known of these corre-

TABLE 45
Relation Between UOP-Characterization Factor and Chemical Composition of Lubricating Oils.

SUBSTANCE	UOP—Characterization Factor Det. From				
	Boiling Pt.*	Mol. Wt.	Viscosity†	Anil. Pt.	Waterman
Fraction A-C	12.3	12.15	12.2	12.3	12.3
Fraction A-Res.	12.85	12.75	12.7	12.4	12.63
Fraction C-1 (arom.)	12.0	12.0	12.0	11.4?	11.7!
Fraction C-Res.	12.9	12.8	12.8	12.3?	12.76
Fraction F-1 (arom.)	12.5	12.4	12.3	12.1	11.8!
Fraction F-Res.	12.95	12.8	12.8	12.7	12.8
Extract A-1‡	9.9	10.0	10.1	....	....
Extract A-17	10.85	10.95	11.05	....	10.3!
Extract B-12	10.35	10.45	10.60	....	(10.7)
Extract B-12H	11.35	11.35	11.55	11.6	11.55
6,11 din-Amyl hexadecane	13.0	12.92	12.9	12.65	13.2
11-Decyl heneicosane	12.9	13.1	13.0	....	13.2
Tri (cyclohexyl-ethyl) methane	11.72	11.72	12.05!	11.8	11.32!
Tri (ethyl benzene) methane	10.55	10.7	10.3!	....	10.0!

* Extrapolated from the 1 mm. data to atomspheric pressures by Lederer's method, Petroleum Z. 31, No. 16 (1935).

† It is remarkable to note that for all the lube fraction tested it makes no difference whether one determines K from the viscosity at 100 °F. or at 210 °F.!

‡ Calculated from the data of Mair et al, J. Res. Natl. Bur. Standards 21, 535, 56 (1938).

lations is the one by Watson and coll.[166] which was built around the UOP-Characterization Factor K defined as:

$$K = \frac{T_B^{1/3}}{d_4^{15.5}}$$

where T_B is the "molar average" boiling point in degrees Rankine at 760 mm and $d_4^{15.5}$ the density of the petroleum fraction under study.

The numerical value of K ranges from about 10 for highly aromatic oils to ~13 for completely saturated hydrocarbons. As the boiling point of most lubricating oils can only be ascertained by rather far extrapolation, K can be determined from Nelson's correlation charts from density and melocular weight or density and viscosity, V-I and aniline point. There has recently been added a method to calculate K from data of the Waterman analysis[170] according to a linear formula:

$$K = 0.086A + 0.106N + 0.132P \qquad \begin{matrix} \pm 0.07 \\ \Delta_{max} = 0.2 \end{matrix}$$

where A,N,P are percent carbon in aromatic, naphthenic and paraffinic bond respectively.

In view of their empirical nature, the reliability of these correlations, as brought out in Table 45, is quite remarkable. There is, however, one fact easily observed—and the writer likes to attach much significance to it—that only among petroleum fractions can these correlations be made to work. On many synthetic (pure) hydrocarbons and synthetic lubricating oils (Fischer-Tropsch oils) the K correlations become entirely inapplicable. May it perhaps be that among all the possible hydrocarbon isomers of one class only those occur in petroleum which fit into these empirical correlation systems? If this "selection rule" could be verified, a powerful new tool could be added to our arsenal of instruments in the search for the composition and origin of petroleum.

In the previous paragraphs the hydrocarbon components of lubricating oils alone have been discussed as if there were hardly any other compounds contained in petroleum-derived lubricating oils. Nothing could be farther from the truth. There occur both in the United States (along the Gulf Coast) and in Russia lube oils containing up to 5 percent naphthenic acid, not to speak of the well known Smackover crudes, the lube stocks of which contain 2 to 3 percent sulphur in as yet entirely unknown combination, which if present as compounds of but 1 sulphur atom per molecule would represent 20 or more percent of the entire fraction. The resins, the chemical composition and physical properties of which have been dealth with at length in Sections III and IV of this series, are probably sulphur and oxygen compounds of cyclic hydrocarbons, which occur in considerable concentrations, (10 to 20 percent) in heavy lube oils and especially in lubricating oils of residual origin. They seem closely related to similar materials occurring in bitumina and are generally considered "intermediate" in composition between hydrocarbons and asphaltenes. As there is increasing evidence that the bitumina are the origin of petroleum hydrocarbons, and not vice versa as had been believed before, it is quite likely that more intense investigations of the chemical nature of these resinous compounds will be undertaken in the near future. So far only the catalytic cracker and the lubricating grease manufacturer know about the presence of nitrogen compounds in some of the higher-boiling petroleum fractions, and they are usually not very enthused about the effects of these nitrogenous materials, but academical information is entirely wanting.

Summary

The so-called paraffinic oils contain mainly hydrocarbons with one to two naphthene rings or possibly a naphthene and an aromatic ring in the same molecule, probably alkylated with a long paraffin chain; normal or iso-paraffines are almost certainly absent (!) Solvent raffinates seem to contain only naphthenes. The so-called "asphalt-base" oils, especially their solvent extracts, consist of condensed aromatic and naphthenic rings with relatively short side chains, have often up to five rings per molecule, and there is much evidence that aromatic and naphthenic rings are often simultaneously contained in the same molecule.[171] Many coastal oils of low V.I. seem to consist almost entirely of condensed ring naphthenes with up to four rings per molecule. As synthetic hydrocarbons of such highly cyclic nature have not yet been prepared and the above data are based solely on statistical analysis, there is still some uncertainty as to the structure of the multi-cyclic hydrocarbons in solvent extracts and low V-I oils. This cannot be said for the high V-I. oils for which statistical analysis and synthetic hydrocarbon data have been found in excellent agreement. The composition of non-hydrocarbon in lubricating oils is still unknown (except perhaps that of the naphthenic acids).

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Simple Automatic Composite Sampling

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Root Petroleum Company

Sampling of liquid and vapor streams in the refining industry is conducted for several reasons, including

1. Maintaining product specifications.
2. Determining magnitude of losses of valuable components into less valuable streams.
3. Determining efficiencies of conversion processes.
4. Determining effect of changes in process variables.
5. Determining average availability of components as charge to contemplated processes.
6. Determining correct bases for payments in interplant sales or purchases, or for payments of royalties.
7. Making component balances.

It is common practice in sampling for maintenance of product specification to obtain "spot" or "grab" samples at regular intervals of one or two hours. Each sample is inspected or analyzed and adjustments made in process variables by the operator if, in his judgment, any changes are indicated. "Spot" samples are considered satisfactory for this purpose since process holdup tends to average compositions in the sampling intervals.

For the remaining purposes listed above it is generally desired to ascertain the average composition of a given stream over a longer period, usually 8, 24 hours, or longer. For these purposes, one method of obtaining average composition is, for a given stream, to obtain a series of spot samples at regular intervals over the sampling period and to mix them in one container. A suitable portion of the composite is then analyzed to obtain the average composition or properties of the stream over the period of the composite. While process holdup tends to average compositions between spot samples, this method usually makes no allowance for changes in rate of flow, since the spot samples for the composite are of equal volume. This factor is usually not serious since, except during upsets, most refinery streams are quite uniform in rate of flow over considerable periods. However, when composite sampling is made routine, operators whose duty it is to prepare the composites are apt to be occupied with operating problems at sampling times, are apt to forget to draw some of the samples, or, as has been admitted by some operators, may draw at one time all of the spot samples required for the composites during the shift.

In order to avoid such uncertainties and to avoid increasing the duties of operating personnel, an automatic composite sampler was developed. This device automatically obtains composite samples by water displacement, and is adjustable with respect to frequency of sampling and to volume of individual samples. Several of these samplers have been in successful operation during the past three years. As examples of the versatility of the instrument, composite samples as follows have been obtained:

Automatic sampler with case removed. The sampler includes a clock-actuated trigger mechanism arranged so that individual samples can be collected in the composite sample container, shown below, at any selected time interval, their volume being controlled by water displacement from the container through a measuring device.

1. Weekly five-gallon composites of catalytic-cracking-unit gas.
2. Weekly five-gallon composites of catalytic-cracking-unit distillate.
3. Weekly five-gallon composites of deethanizer-overhead gas.
4. Weekly five-gallon composites of stabilizer-overhead gas.
5. Weekly three-gallon composites of alkylation-unit B-B charge.
6. Weekly five-gallon composites of alkylate.
7. Weekly three-gallon composites of debutanizer overhead.
8. Daily one-gallon composites of isomerization-unit charge.
9. Daily one-gallon composites of isomerization-unit effluent.
10. Daily one-gallon composites of straight-run gasoline.

In addition to several other regular composites, numerous 24-hour composites of one to five gallons have been obtained during special tests of process equipment.

As a more recent development, a modification of the automatic sampler will be installed for sampling the effluent of a gasoline treating plant. Since this treating plant does not operate continuously, the sampler to be installed is provided with means for sampling proportional to flow.

Velocity Sizing of Water Lines

G. R. ADAMS

VELOCITY sizing of water lines is a quick and economical method of correctly determining piping needs inside coolers, condensers, and service-water connections.

The curves on the next page are a graphical solution that quickly indicate the water velocity for any desired gallons per minute flowing quantity in addition to the usual friction head losses.

Sizing Specifications

Water-line specifications will vary for different operators and contractors. Lines of four-inch and less usually have a 2.5-foot head-loss per 100 feet, while lines over 4 inches in size have 5 or even 4 feet per second velocity limitations. Some engine manufacturers specify a 4-foot-per-second maximum velocity for piping on their cooling units. A heavyweight dashed-line is drawn on the curves at the 2.5-feet-per-100-feet head-loss and the four-feet-per-second velocity lines.

The regular-weight dashed-line curves are the usual gallons per minute flowing rate versus the head-loss for each size of pipe, while the regular-weight solid-line curves are the gallons per minute flowing rate versus velocity for each size of line.

Use of the Curves

As an example in using the curves, find the correct size of pipe for 40, 100, 400, and 1000 gallons per minute flowing rates. Keeping near or below the heavyweight dashed line, a 2½-inch pipe will be satisfactory for 40 gallons per minute; a 4-inch line will be more than sufficient for 100 gallons per minute; a 6-inch, for all practical purposes, will be large enough for 400 gallons per minute; and a 10-inch line will be correct for 1000 gallons per minute flowing conditions. To find flowing velocities from the curves, the values are read for each gallon per minute rate on the desired line size. For example at a 400-gallons-per-minute flowing rate, the velocity in an 8-inch line is 2.6 feet per second, a 6-inch pipe will have 4.4 feet per second, and a 4-inch line will have 10.1 feet per second.

Head-Loss Calculations

If the lines are very long or engineering requirements are of such nature that a detailed head-loss is required, then this actual loss may be calculated as follows:

A. From the friction values at the left hand side of the curve sheet;

B. As described in "Pumps" by Kristal and Annett in their chapter on economical pipe sizes for pumps; or

C. From the "Cameron Hydraulic Data" reference handbook by Shaw and Loomis in their chapter on Water Data.

Head-loss calculations are made by using Williams and Hazen's empirical formula as given in the Cameron Hydraulic Data reference handbook.

$$1.\quad f = 0.2083 \times \left(\frac{100}{C}\right)^{1.85} \times \frac{q^{1.85}}{d^{4.8655}}$$

in which

f = friction head in feet of liquid per 100 feet of pipe.
C = Constant accounting for surface roughness.
q = flow in gallons per minute.
d = inside diameter of pipe in inches.

and

v = velocity in feet per second or the feet of line that the liquid fills each second.

The friction head-loss when "C" is 100 for welded and seamless steel pipe; "q" is 100 gallons per minute flowing rate; and "d" is 4.026 inches for standard weight steel pipe; will be

$$2.\quad f = 0.2083 \times \left(\frac{100}{100}\right)^{1.85} \times \frac{(100)^{1.85}}{(4.026)^{4.8655}}$$

$$3.\quad f = 0.2083 \times 1 \times \frac{5012}{877.0} = 1.190 \text{ feet per 100 feet of pipe.}$$

Velocity Calculations

Velocity points for plotting are calculated as follows:

$$4.\quad v = \frac{q \times (2 \times 12)^2}{7.48 \times 60 \times \pi \times d^2} = \frac{q}{2.448 d^2}$$

or

$$5.\quad q = 2.448 d^2 v$$

where q, d, and v are the same as under equation 1.

A four-inch line with "d" equal to 4.026 inches and a 4-foot per second velocity "v" will have

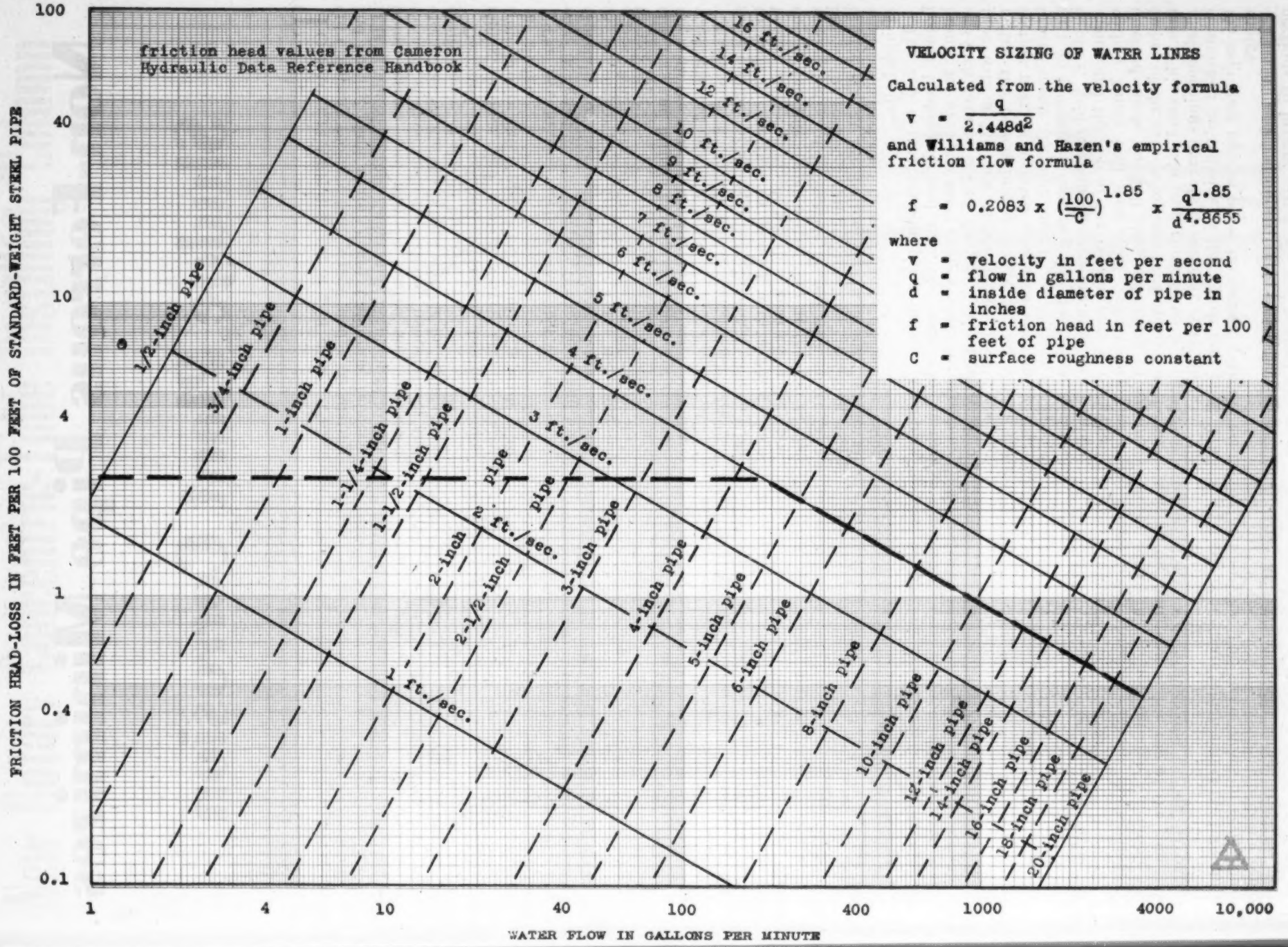
$$6.\quad q = 2.448 \times (4.026)^2 \times 4.00 = 158.7 \text{ GPM.}$$

hence, the 4-foot-per-second-velocity line intersects the 4-inch pipe gallons per minute versus head-loss line at the 158.7 gallons per minute flowing rate. The other points in Table 1 were calculated and plotted in the same manner.

TABLE 1
Velocity Versus G.P.M. Flowing Rates

Velocity in Feet per Second	G.P.M. FLOWING RATES			
	1-Inch Pipe $D^2 = 1.1004$	4-Inch Pipe $D^2 = 16.209$	10-Inch Pipe $D^2 = 100.40$	20-Inch Pipe $D^2 = 367.87$
1	2.69	39.68	245.8	901
2	5.39	79.35	491.5	1801
3	8.08	119.0	737.3	2701
4	10.77	158.7	983.0	3602
5	13.47	198.4	1229	4502
6	16.16	238.1	1475	5403
7	18.85	277.7	1720	6303
6	21.55	317.4	1966	7204
9	24.24	357.1	2212	8105
10	26.94	396.8	2458	9005
12	32.32	476.1	2949	10810
14	37.71	555.5	3441	12610
16	43.10	634.8	3932	14410
18	48.48	714.2	4424	16210
20	53.87	793.5	4915	18010

WATER FLOW IN GALLONS PER MINUTE


friction head values from Cameron Hydraulic Data Reference Handbook
VELOCITY SIZING OF WATER LINES
Calculated from the velocity formula
v = q / 2.448d²
and Williams and Hazen's empirical friction flow formula
f = 0.2083 x (100/C)^1.85 x q^1.85 / d^4.8655
where
v = velocity in feet per second
q = flow in gallons per minute
d = inside diameter of pipe in inches
f = friction head in feet per 100 feet of pipe
C = surface roughness constant
FRICTION HEAD-LOSS IN FEET PER 100 FEET OF STANDARD-WEIGHT STEEL PIPE
WATER FLOW IN GALLONS PER MINUTE
100
40
10
4
1
0.4
0.1
1
4
10
40
100
400
1000
4000
10,000
16 ft./sec.
14 ft./sec.
12 ft./sec.
10 ft./sec.
9 ft./sec.
8 ft./sec.
7 ft./sec.
6 ft./sec.
5 ft./sec.
4 ft./sec.
3 ft./sec.
2 ft./sec.
1 ft./sec.
1/2-inch pipe
3/4-inch pipe
1-inch pipe
1-1/4-inch pipe
1-1/2-inch pipe
2-inch pipe
2-1/2-inch pipe
3-inch pipe
4-inch pipe
5-inch pipe
6-inch pipe
8-inch pipe
10-inch pipe
12-inch pipe
14-inch pipe
16-inch pipe
18-inch pipe
20-inch pipe

Non-Ferrous Pipe Minimizes Sludge Plant Corrosion

TO FACILITATE disposal of acid sludge, Union Oil Company recently built a new sludge circulating and heating plant at its Oleum, California, refinery. It is arranged so that sludge may be pumped to any point desired, and may be heated and circulated through storage tanks to maintain temperatures and viscosities within the ranges suitable for satisfactory performance of sludge burners.

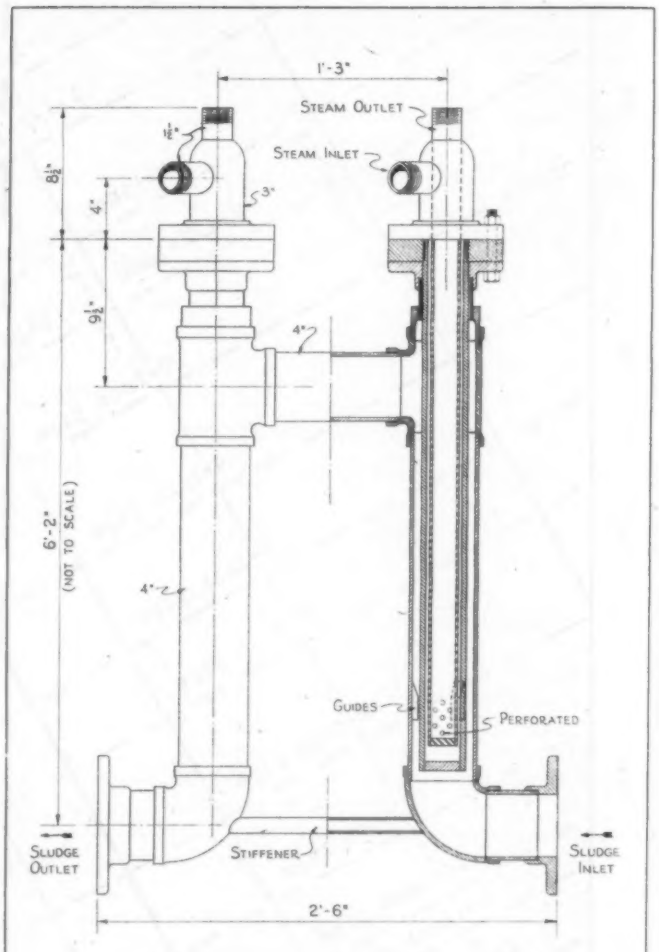
Designed to combat corrosion, heating units, piping, fittings and control valves are fabricated from bronze and copper where contact is made with the sludge.

The five heating sections included in the plant were designed by the company's engineers and built especially for this installation. Each section consists of two similar parts, arranged to carry the sludge in series flow throughout the assembly. Four-inch bronze pipe is used in the construction, with bronze ells, tees and fittings having slip joints for connecting with the pipe by brazing with silver to eliminate washouts and incipient leaks.

Each part of the section is constructed with the same number of fittings, but assembled back-to-back to form the section by joining with a 9¼ x 4-inch bronze plain-end nipple which is inserted in the open end of the side outlet of the tees at the top of the section parts, and, likewise brazed with silver. The legs of the section are approximately 5 feet long with bronze ells at the bottom and a bronze nipple to which is attached a bronze flange drilled for eight ¾-inch bolts.

The top of the section parts have a 4 x 3-inch bronze bushing brazed into the open end of the bronze tee to take a short copper nipple for attaching a 3-inch bronze bolted flange. A blind-end carbon tube with a 7½-inch carbon flange, also drilled to match the 3-inch bronze flange at the top of the section part, is inserted into each leg. Bronze lugs brazed inside the pipe legs 4 inches from the lower part on 120-degree centers guide the carbon bayonet in place. Acid-resistant gaskets are used on all flange joints, and the top of the section part is completed with a 3-inch bull plug. A 1½-inch steel pipe is inserted inside the bull plug, extending to within 2 inches of the carbon bayonet when placed in the section. This steel tube is perforated with a number of small openings near its closed end. A 1½-inch nipple is attached on the side of the 3-inch bull plug. The 1½-inch steel pipe is never in contact with the acid sludge.

The five units are installed in series, having connections to bronze manifolds and control valves so that the sludge may be pumped to any point desired, or circulated through storage tanks to maintain required temperature and viscosity.



Sludge heater section.

Steam flows through each of the sections. The steam enters the section through the nipple in the side of the bull plug at the top of the connections, passes downward through the carbon bayonet and leaves through the perforated steel tube, carrying with it any condensate which enters the tube through the perforations. The steam flows in series through each part of the sections, condensate being removed at intervals through small steam traps to prevent over accumulation of water in the last section of the series.

New Rotary Concentric-Tube Distilling Column

A NEW rotary concentric-tube distilling column, having a very high efficiency factor for fractional distillation, has been developed at the National Bureau of Standards by Charles B. Willingham, Vincent A. Sedlak, James W. Westhaver, and Frederick D. Rossini, in connection with the cooperative research program on hydrocarbons sponsored jointly by the bureau and the American Petroleum Institute.

The demand for fuels providing greater efficiency, higher speeds, and more concentrated power, as well as development of various devices making use of new types of hydrocarbons, has resulted in a transition in the petroleum industry from the manufacture of broad fractions toward the production of narrower fractions and even pure compounds. The experimental program now under way at the National Bureau of Standards has two objectives: the investigation of the chemical constituents of the crude oil based upon the actual isolation of pure hydrocarbons; and securing of high-purity standard samples for the calibration of instruments used in analyzing such complex mixtures as aviation fuels and synthetic rubber components. The new distilling column, which is expected to have wide application to fractionation processes, is expected to aid materially in the bureau's hydrocarbon research program.

Increasing Throughput

Numerous devices, based on both theory and experiment, are available for producing high separating efficiencies in distilling columns. Among these are open tubes of small diameter, concentric tubes with a small annular space, and parallel plates with a small space between them. All, however, are limited in use by a relatively low throughput, that is, quantity of material that may be volatilized per hour. According to theory, the efficiency may be improved by decreasing the spacing of the tubes or plates of the column, by decreasing the throughput, or by increasing the rate of diffusion of the gas molecules through the distilling column. Decrease in the spacing (or diameter) has been carried about as far as practicable, while decreasing the throughput below the present low values is not feasible. Moreover, in a static

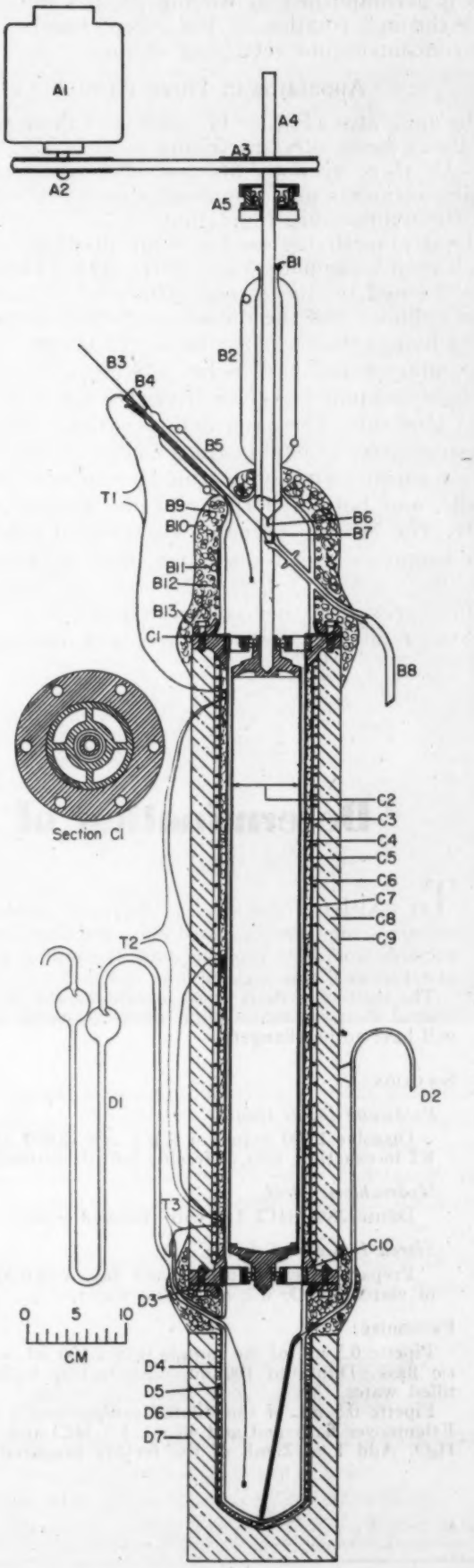


FIGURE 1

Schematic diagram of the rotary concentric-tube distilling column recently developed at the National Bureau of Standards. A1, A2, A3, A4, A5 are parts of the motor assembly for rotation of the inner column. The principal parts of the head (B) are: head condenser (B2) with water jacket; control (B3) for valve (B7), used for sampling liquid reflux in head; side condenser (B5); glass rod (B6) to direct flow of liquid reflux onto valve; outlet (B8) for head sample; thermal insulation (B9); aluminum foil (B10); and nichrome heater unit (B12). The steel rectifying section (C) consists of the cylindrical rotor (C2), concentric with the open tube (C4); the nichrome heating elements (C7); thermal insulation (C8); and aluminum foil (C9). The more important parts of the pot (D) are the butyl carbitol manometer (D1), the tube (D2) for withdrawing samples of material, and the pot heater (D5).

apparatus, for a given temperature and composition, the rate of diffusion of the gaseous molecules is substantially constant.

Accordingly, the new distilling column was designed to improve separating efficiency by increasing the diffusion rate of the molecules in the gas phase. This is accomplished by forcing the gas into turbulence through rotation of the inner closed cylinder in a concentric-tube rectifying section.

Apparatus in Three Parts

The apparatus (Figure 1) consists of three parts—the Pyrex head, steel rectifying section, and Pyrex pot. All three sections are provided with external heating elements and copper-constantan thermocouples for temperature regulation.

The steel rectifying section of this distilling column is the empty annular space, 0.043 inch (1.09 mm.) wide, formed by the inside surface of a stationary outer cylinder and the outside surface of a rotating closed inner cylinder, 2.928 inches (7.44 cm.) in outside diameter and 23.0 inches (58.4 cm.) in length. A motor-and-pulley system drives the rotor at speeds up to 4000 rpm. The outer cylinder is enclosed in an asbestos-covered metal heating jacket surrounded by three separate nichrome heating elements for the top, middle, and bottom portions of the jacket, respectively. The heating elements are covered externally with magnesia insulation and a layer of aluminum foil.

The Pyrex head consists principally of a water-jacketed condenser directly above and opening into an electrically-heated chamber, in which the liquid reflux may be collected and sampled by means of a glass valve. The heated chamber is surrounded by asbestos wool insulation and aluminum foil.

The pot, made of a three-inch Pyrex pipe, is sealed at one end and provided with a butyl carbitol manometer and a tube for withdrawing samples. Heated externally by a Glas-col special sleeve type heater, it is surrounded by magnesia insulation covered with aluminum foil.

Thermoelements

Three thermoelements are provided. One measures the difference between the temperature of the top portion of the rectifying section and the liquid-vapor equilibrium in the head. Another measures the difference between the temperature of the middle portion and the mean temperature of the top and bottom portions of the rectifying section. A third is used to determine the difference between the temperature of the bottom of the rectifying section and the temperature of the liquid in the pot.

For high values of throughput—two to four liters of liquid per hour—this distillation column, when operated at 4000 rpm, has an efficiency factor about ten times those previously reported for other rectifying columns. The efficiency factor changes relatively little with throughput at a given speed rotation, but increases markedly with speed of rotation. This column also has low values of pressure drop per unit throughput, which may be quite advantageous for distillations at low pressure.

Determination of Sulphur in Caustic Scrub

TO RAPIDLY evaluate the degree of saturation of caustic scrub solutions used for scrubbing hydrogen sulphide from light products such as gasoline, naphtha, and kerosine, this method was devised.

The method is rapid and accurate enough to predict several hours ahead of time when the scrub solution will have to be changed.

SOLUTION:

Potassium Iodide-Iodate.

Dissolve 1,000 grams of KIO_3 and 12,000 grams of KI in exactly 1 liter of freshly boiled, distilled water.

Hydrochloric Acid.

Dilute 38% HCl 1:1 with distilled water.

Starch Indicator Solution.

Prepare a fresh solution every day. Add 0.25 grams of starch to 100 ml. of boiling water.

PROCEDURE:

Pipette 0.5 ml. of the sample into a 100 ml. volumetric flask. Dilute to 100 ml. with freshly boiled, distilled water.

Pipette 0.5 ml. of the diluted sample into a 500 ml. Erlenmeyer flask and add 40 ml. 1:1 HCl and 100 ml. H_2O. Add 1 to 2 ml. of the freshly prepared starch indicator solution and titrate with the Potassimum Iodide-Iodate solution to a pale blue color.

CALCULATION:

ml. KIO_3-KI x 0.07462 = Pounds S per gallon.

NOTES:

All solutions should be kept as cool as possible to prevent evolution of H_2S.

Samples should be diluted as directed to avoid loss of H_2S.

It may be necessary to further dilute samples containing very large amounts of sulphur, hence changing the calculations.

Samples should be taken in glass-stoppered bottles or bottles with very tight-fitting corks to prevent any possible loss of H_2S before the sample is tested.

The above operation should be conducted as rapidly as possible.

In desulphurizing equipment where mercaptan sulphur is being converted to H_2S and the H_2S scrubbed from the gasoline with NaOH solutions, it is possible to predict within a half hour, 6 or 8 hours in advance the necessity of changing scrub solutions. Consequently keeping the processing men warned in advance.

REFERENCE: Text book of Quantitative Inorganic Analysis. Kolthoff & Sandell.

FOR CONDENSER TUBES

MOST plants operating a number of condensers, heat exchangers and similar equipment using condenser tubes find that tube life varies, a bundle in one condenser lasting longer than in another. This points up the fact that obtaining longer life hinges on detailed study of individual conditions, such as Revere is ready to make for you.

Take the case of a shell-type condenser following a high stage compressor. Admiralty tubes lasted about a year, red brass less, and treatment of the gas had produced no improvement. Failure was due to thinning at the cool end, and cracking at the hot end. Microphotographs taken in the Revere Laboratory showed transgranular corrosion-fatigue cracks originating from the base of the pits on the vapor side of the tube. Hence it was evident that failure was due to a combination of corrosion and vibration.

The remedies recommended were: redesign of inlets and baffles, reduction of vibration in adjoining machines, bracing of the condenser, and alkali treatment to raise the pH up to 7.5—8.5. The result: substantially increased service from inhibited Admiralty tubes.

Revere stands ready at all times to work with tube users in the endeavor to extend the period between re-tubings.

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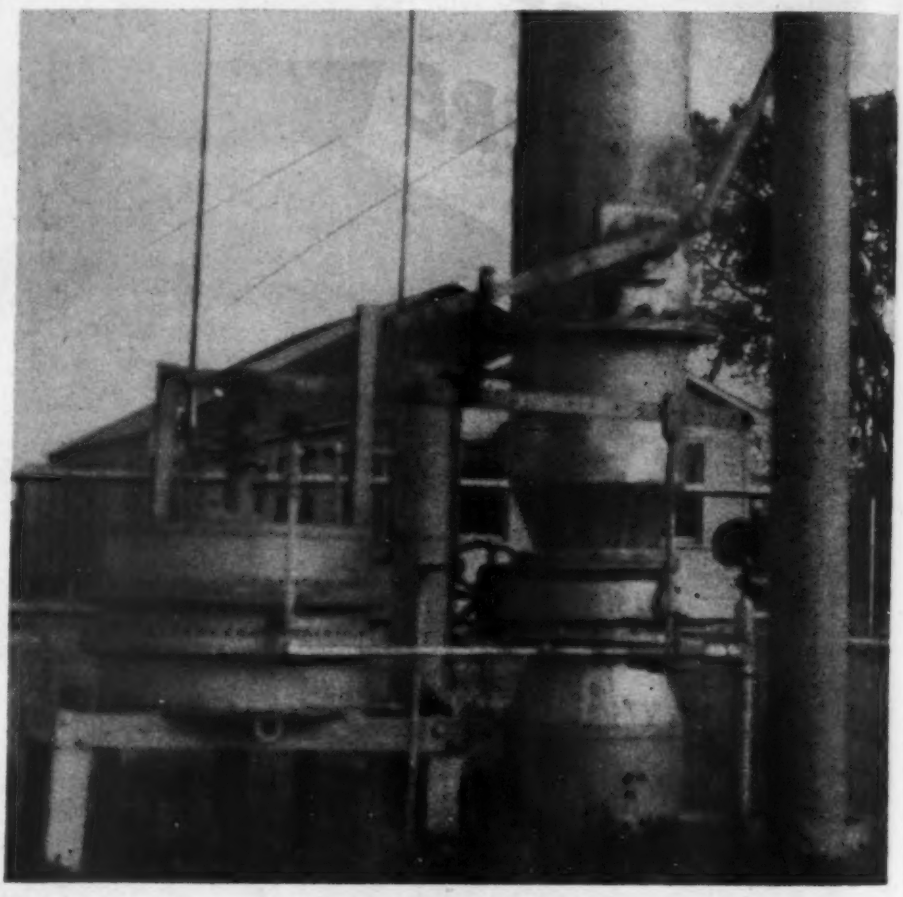
OPERATION HOW TO DO IT MAINTENANCE

Propane Storage Tanks Made of 26-Inch Pipe

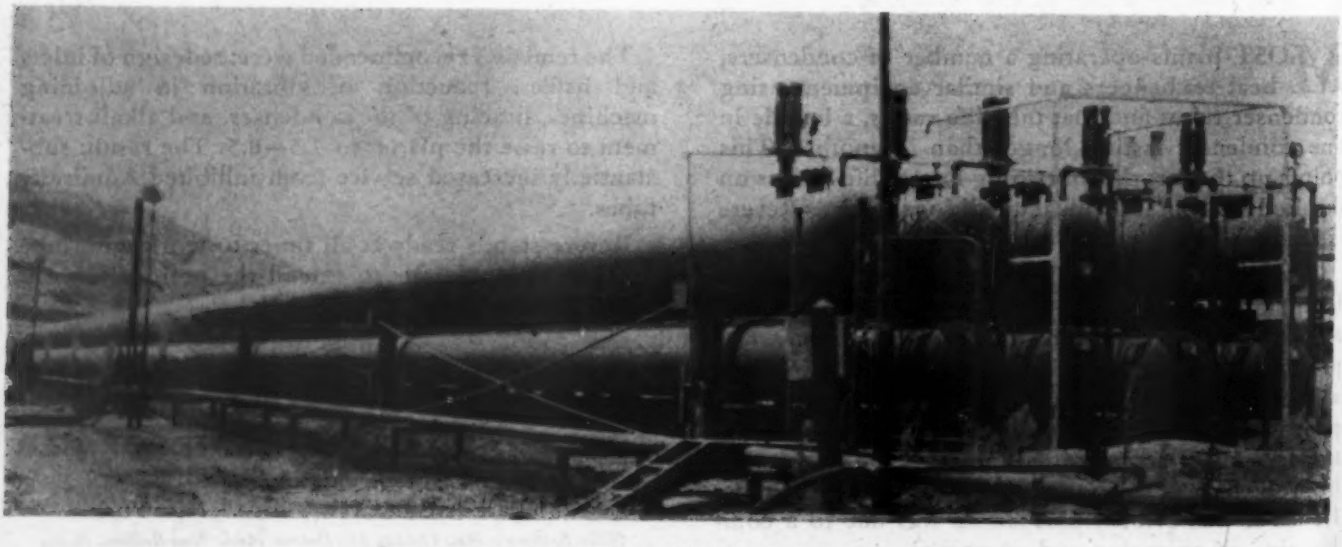
DEL VALLE Gasoline Company, in overcoming a shortage of steel for fabrication of the customary pressure vessels in California, fabricated a satisfactory battery of propane-storage tanks from 26-inch pipe. The pipe is made from ¼-inch steel with a longitudinal welded seam, with heads of ⅜-inch steel. The battery includes 10 cylinders, each 280 feet long—end-to-end the tanks are more than half a mile long. The tanks have a working pressure of 285 psig at 150° F.

The installation problem was solved by pouring concrete supports at intervals. Supports are reinforced with steel on the top surface, and with vertical standards at each end of the piers.

The tanks are set in pairs, one above the other. The lower tank rests upon the concrete pier while the upper rests upon a steel cross member, in turn supported by vertical standards. Steel pads welded to the lower surface of each tank serve as the contact between the tanks and the piers for the lower row, and the steel cross member for the upper row. The pads are sections of channel steel, attached to the tanks with the channel facing the tank shell, reinforced with strap steel welded to each side at the angle on the outer side. Similar sections of channel steel are attached to the concrete pier and to the steel cross member in such position that the outside of the pad on the tank nests into the channel of the section attached to the concrete and to the cross member. The stationary pads on the supports have roller bearings made of round bar steel which separate the two channel-iron sections, to permit longitudinal movement. Alternate side vertical standards extend to the side of the top vessel. Posts with shoes guide the tanks to prevent lateral movement, and force the tank to move lengthwise when expanding or contracing. Vertical posts are set between the rows of vessels with guides to keep the inside rows horizontal and in line with the longitudinal creep. The tanks being anchored at the end equipped for filling and pumping out, guides and roller bear-

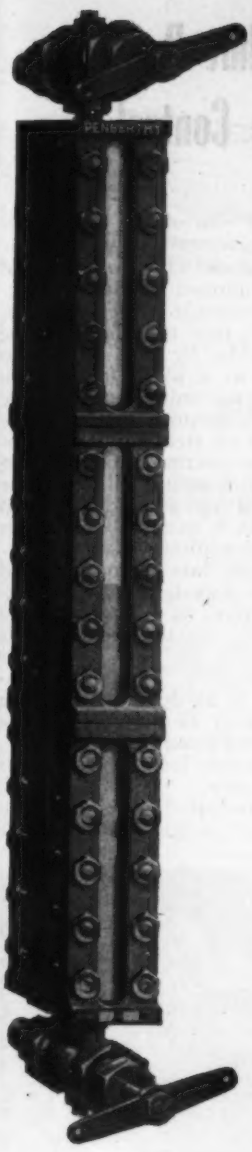


The pressure controller shown in the photograph was built in a Michigan plant's shop, to regulate the pressure on the incoming gas line. It consists of a drum-type case with welded flanges and a double diaphragm. The shaft from the diaphragm extends from the top of the case through a packing gland to the actuating lever controlling the damper-type valve in the vertical section of the line. Vacuum or pressure can be controlled by switching connections in the diaphragm case.



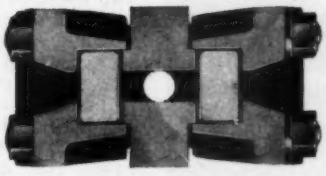
Propane storage tanks made of eight 26-inch pipes, 280 feet long.

FOR EVERY GAGE REQUIREMENT



Whatever your liquid level gage requirements, there is a Penberthy Gage that will meet your needs. These gages are suitable for the various pressure and temperature conditions of the oil industry. All Penberthy Gages conform with A.P.I.-A.S.M.E. requirements.

Write for a copy of Catalog 34-A.



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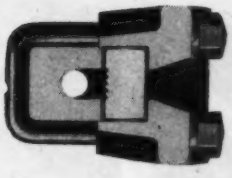
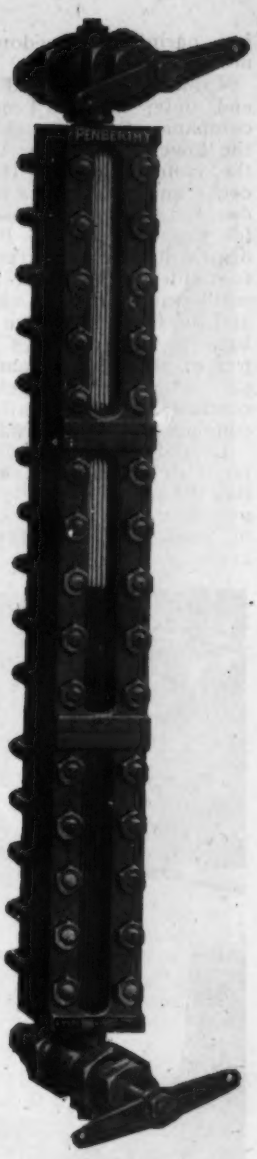
DROP FORGED STEEL LIQUID LEVEL GAGES

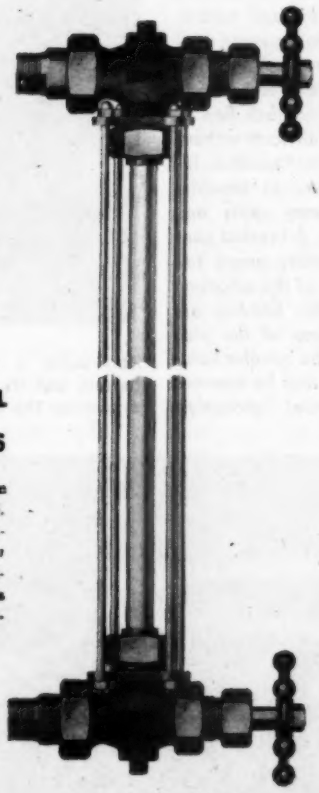
Used to observe color and density of liquids under high pressures, and/or temperatures. Construction is exceptionally rugged . . . . similar to Reflex types.



PENBERTHY *Reflex*
DROP FORGED STEEL LIQUID LEVEL GAGES

Liquid shows black — empty space shows white. Preferred wherever liquid level must be easily and positively visible . . . and when liquids are under high pressure or at high temperature.





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Made of Chromium-molybdenum alloy temperature-resisting steel, extra heavy throughout. Stainless steel trimmed. Tubular glass type gages also available in various other metals suitable for practically all conditions.

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Water shows black — steam shows white. U-Bolt construction is strongest and simplest to service. Glass replaced by simply removing nuts on face of gage . . . unnecessary to work between gage and boiler.

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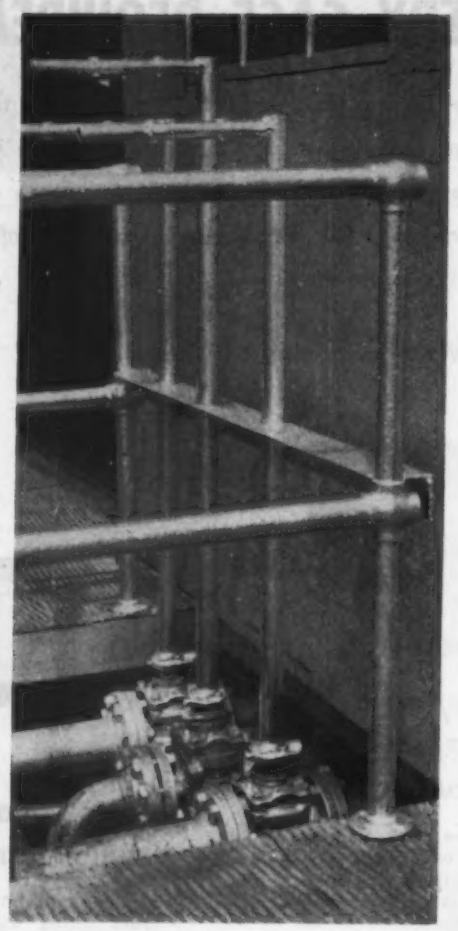
ings permit full freedom of longitudinal movement.

Tanks are connected at the operating end only, having 4-inch nipples with companion flanges so that liquid entering the lower of the pair will rise through the connection into the upper. Gauge cocks and glasses are installed in pairs, one for the lower vessel and the other for the one on top. Filling and withdrawal headers are attached to the bottom side of the lower tank in the pair, while vapor lines to equalize the tanks and the truck tanks are at the top of the battery. Safety valves are installed on top of the upper member of the tank pair, attached to 4-inch nipples having companion flanges similar to those which connect the tanks in pairs.

Coated with heat reflecting paint, the tanks do not absorb sufficient temperature to cause difficulty in accumulated pressure. In order to assure evacuation of fluid, the tanks are installed on a grade of 1½ inches to 280 feet.

Forms for concrete piers can be made from non-returnable steel drums with the heads removed. Foundation bolts are installed in the usual manner. The drum steel can be left in place, or split away as desired. During the lumber shortage, this type of form has been particularly useful.



When intake, discharge and relief lines are under the compressor-room floor, and control fittings are outside the building, opening and closing the controls may be simplified with extension handles. The valves in this installation are lubricated plugs, flanged into each line at a point which makes operation difficult without extension handles. But with the handles installed, the operating levers are at shoulder height so that the operator may easily and quickly open or close the valves. A bracket connecting each section of the safety guard rail serves as a support for the risers of the extension handles. The lower ends of the handles are broached to fit the square stems of the plug valves with only the weight of the handles holding them in place, so that they may be removed when necessary to insert special lubrication.

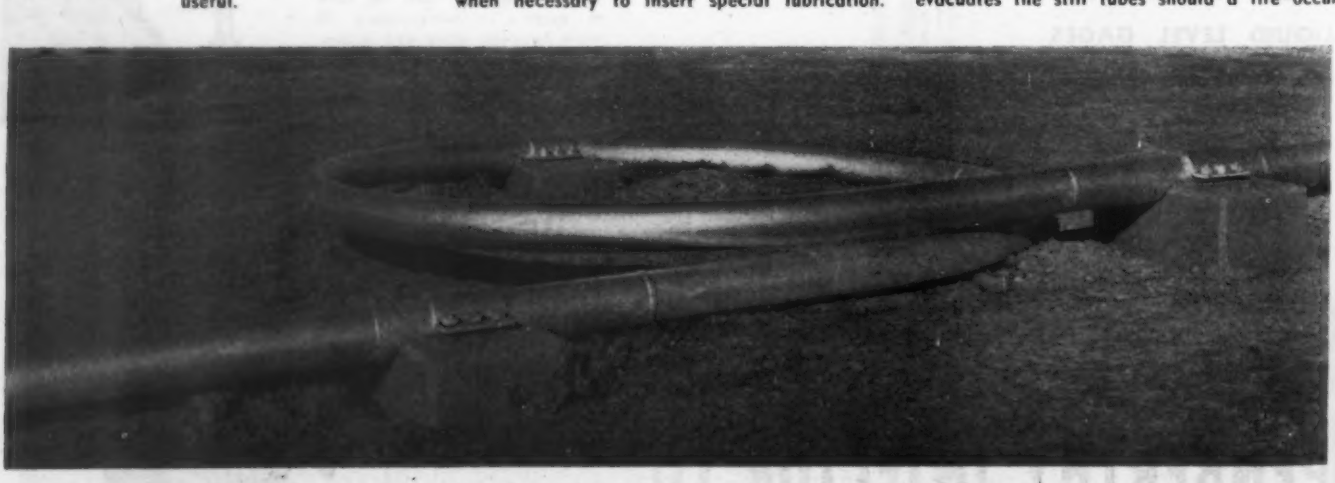
Refinery Shut Down By Remote Control

AN emergency shut-down of a refinery unit may be necessary under some conditions. Hancock Oil Company of California has equipped its plant at Signal Hill with automatic devices and remote controls so that the plant can be shut down quickly by one man who presses buttons at a place far enough from the operating units for complete safety. No one is required to run here and there to shut off steam and fuel controls. The entire operation of shutting down the plant, dropping oil from the heaters, and shutting off the steam at the charge pumps is accomplished from the single control station.

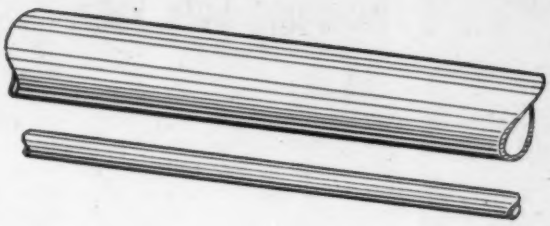
Weather bureau data were consulted when the system was designed to determine the direction of the prevailing winds, so as to install the controls where they would be the safest for workers in the refinery. The control station is mounted in an open shelter on a post so that it may be punched on the run. Wiring is led from this control to shut-down apparatus located in various parts of the refinery.

Steel valves are installed on the drop-out piping of the various heaters in the

This motor is started by pressing the control button, and its operation opens a valve which evacuates the still tubes should a fire occur.



Complete loop in the high-pressure residue gas line of a gasoline plantis used to absorb longitudinal expansion, as shown in the photograph. The six-foot loop is anchored at the top with a clamp to a concrete pier toprevent movement. Line clamps on the base legs of the loop permit longitudinal movement of the line to be absorbed by the loop. Constructionof the loop was simplifed by using five sections of pipe welded together.

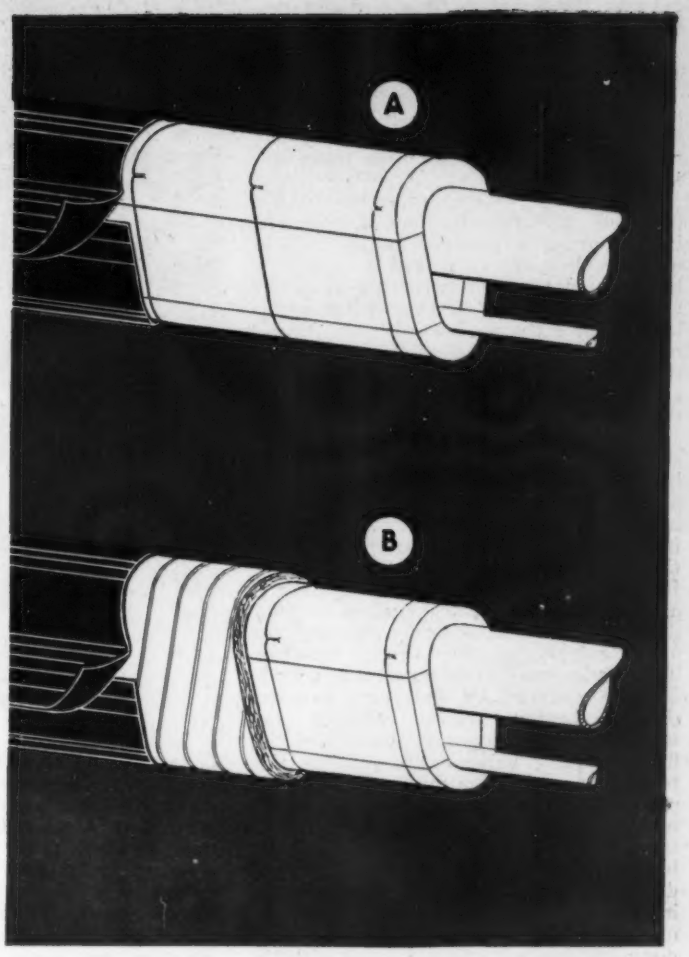


How would YOU insulate these lines?

Here's the problem: A 4" line carrying a high-viscosity processing oil at 130° F. runs overhead from a heated outdoor storage tank across a factory yard to the processing operation. 1½" below it runs a 1" tracer line carrying saturated steam at approximately 15 lbs. pressure (250° F). The plant is located in the Great Lakes region. How would you insulate these lines?

A ☐ Half section of 85% Magnesia pipe covering on top of oil line and another on bottom of steam line. Join the pipe covering with 85% Magnesia block on both sides. All 85% Magnesia to be 1½" thick. Point up joints with asbestos cement and weatherproof.

B ☐ Cover with 85% Magnesia as above, but use standard thickness. Then apply one layer of 1" hair felt and weatherproof.



Here's what the Armstrong engineer advised:

The problem is to maintain the viscosity of the process oil regardless of outside temperatures with a minimum use of steam. Therefore, the insulation must surround both pipes in such a manner that heat can be transferred from the steam line to the oil line. The installation is comparatively permanent and should serve for many years.

Both methods shown are efficient and lasting. Under average service conditions, when winter temperatures do not drop below zero for any appreciable length of time, method (a) will prove entirely satisfactory. But in localities where sub-zero winter temperatures are common, extra precautions have to be taken to guard against thickening of the oil in a line. That's why the Armstrong engineer recommended method (b) for the job in the Great Lakes region. He knew that, while somewhat higher in original cost, extra insulating efficiency furnished by the one inch layer of hair felt would provide inexpensive insurance against production difficulties if the oil congealed in the line in extremely cold weather.

Sometimes it is possible for Armstrong engineers to point out sound methods of heat insulation that give you immediate savings. In other cases, such as this one, their thorough training and long experience indicate that it is better to spend a little more now in order to save larger sums in the long run. When you call in Armstrong's Contract Service—for a bid, for a complete job, or just for informal advice—you can count on getting recommendations that will genuinely serve *your* interest.

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Complete Contract Service For All Temperatures

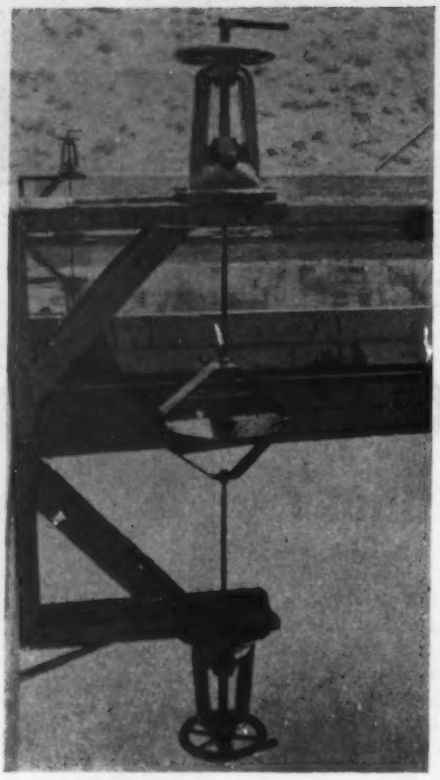
From 300° Below Zero

To 2600° Fahrenheit

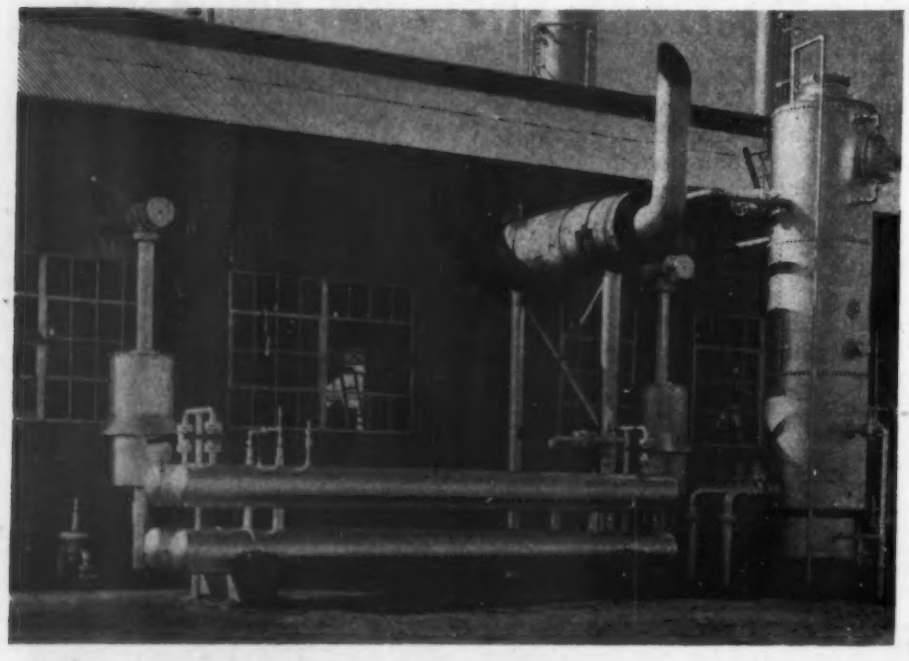
plant, at the most convenient point for the installation of automatic operating devices. These valves have rising stems, and the hand wheels are replaced with sprockets for chain operation. Electric motors with speed-reducing gear-boxes are placed on brackets for alignment of the driven sprocket of the motor and the sprocket on the stem of the drop-out valve. Roller chain is used for transmission of power.

To stop the motor connected to the drop-out valve after it opens the valve to full capacity, a positive stop-switch is used. A bracket is placed to carry a button actuated stop-switch to break the current, the button of this switch being located directly in line with the travel of the valve rising stem, so that when the valve is full open, the end of the stem pushes against the button to break the circuit and stop the motor.

Two supplemental devices are installed for additional protection of the heater. One is a motor-operated valve system similar to that used for the drop-out valve, which is installed on the line from the heater charge pumps to divert the flow of oil from the tubes of the furnace to a blowdown, and the other is a motor-operated valve system to shut down the hot-oil pumps by stopping the steam supply to their driver turbines. The device used consists of a magnetic switch, operated by the same remote-control button which actuates the motors on the drop-out valves, and the flow-diverting valve on the charge line to the furnaces. This magnetic switch is placed near the steam-supply lines to the turbines, and when the coil is energized the automatic steam control on the turbines are tripped and the turbines shut down at once. With these safety devices installed, any person may press the remote control and shut the operation down quickly without danger to himself, or others.



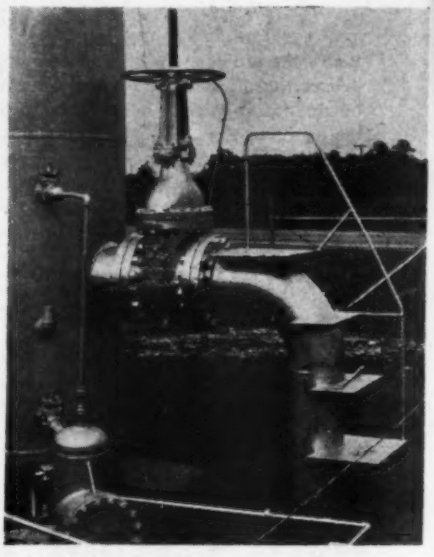
Close skimming of oil on water drained from equipment can be performed with the apparatus shown. A bracket is set to center over the oil outlet pipe. A valve bonnet, handwheel, and stem, are bolted to the bracket for the control mounting. An extension is welded to the valve rising stem to carry a skimmer scoop with an attached nipple which closely fits the oil outlet pipe. A handle brazed or welded to the top of the valve stem serves for adjustment and is locked in place by the valve handwheel.



A space-saving, economical compressed air storage tank was constructed in a gasoline plant's maintenance shop and installed as shown in the photograph. Four 22-foot lengths of 16-inch pipe were used with standard-pipe-size welding caps welded on the end of each length of pipe to complete the pressure vessel. Two of the pipes were set on concrete piers and the remaining two mounted above with 4-inch welded pipe nipples serving as spacers and to connect the pipes. Inlet and outlet headers are shown at each end of the assembled pipes. A pressure gauge has been mounted so that it can be seen by an operator from the door or window of the compressor house. Other fittings provide air for other uses about the plant. Air is stored in the tanks at 300 pounds.

Steps and Guard For Reaching Gate Valve

MANY large gate valves, mounted above the ground, are set with the stem horizontal to aid in wheeling the units for opening or closing, even though in lines where vibration is encountered, such a setting places a large, unbalanced load on the flanges and bolts. One company avoids this unsatisfactory mounting of large valves by setting them so that the stem is vertical, and then utilizing the riser and run of the pipe turn as ladder and platform for the operator as he spins the handwheel.



This arrangement provides steps for reaching the valve and a safety brace while turning it.

In the unit shown, steps are formed from light tank plate with one edge trimmed to fit the curve of the pipe. The steps are then welded to the pipe at proper intervals, with an extra large piece serving as platform at the valve. Addition of a handrail not only stiffens the setup, but also provides the operator a rail to assist him in mounting the unit, and to brace himself while manipulating the valve wheel. If desired, the step plates can be made slip-proof by criss-crossing them with ridges formed by welding beads.

Reinforcing to Protect Gate Valves From Shock

IN CERTAIN localities the stresses resulting from earth tremors must be considered in the design of piping systems, in addition to the more usual problems of thermal expansion, line pressure and temperature, and other service con-

. . . a name that's known internationally for advanced design, engineering and construction of Petroleum Refineries

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G·MV

COMPACTNESS
RELIABILITY
VERSATILITY
ECONOMY

in the smaller

G·MX

Cooper-Bessemer's highly successful V-angle, 2-cycle compressor units are now built in two power cylinder sizes: Type GMX from 250 to 625 hp; Type GMV from 400 to 1000 hp. Both are built in 4, 6, 8, and 10 cylinder sizes rated 62½ and 100 hp per cylinder respectively.

Repeatedly, in hundreds of outstanding installations, the GMV has demonstrated the value of its modern design and its many advanced features. Now, without exception, all these advantages are available in the smaller GMX for those compressor jobs requiring less power but all the stamina, on-stream reliability, and operating economy for which the GMV is noted.

The nearest Cooper-Bessemer office is always ready to cooperate with you or your engineers in investigating and planning the ideal compressor set-up for your present or future needs.

TYPE
G·MV & G·MX
Compressors

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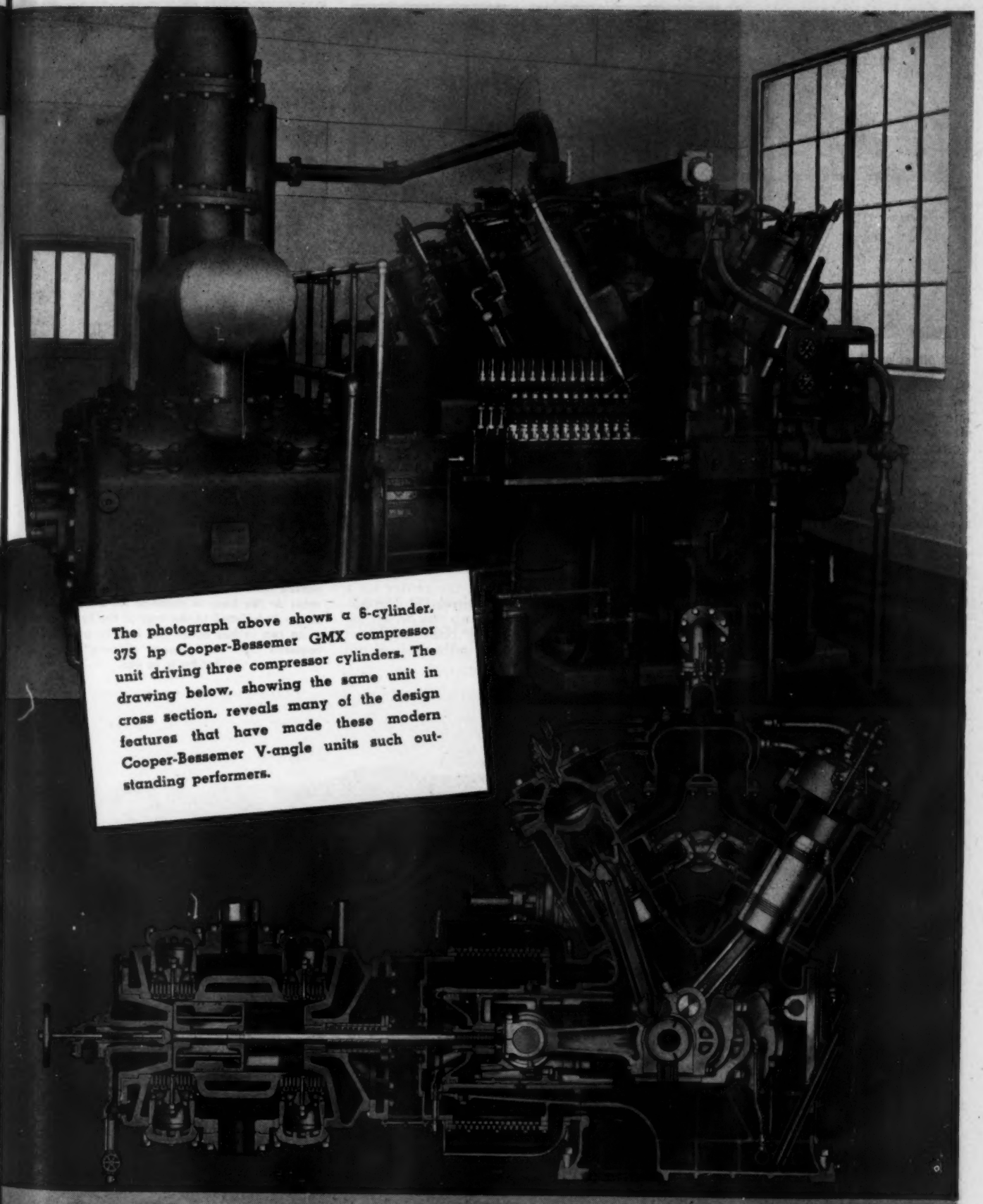

The photograph above shows a 6-cylinder, 375 hp Cooper-Bessemer GMX compressor unit driving three compressor cylinders. The drawing below, showing the same unit in cross section, reveals many of the design features that have made these modern Cooper-Bessemer V-angle units such outstanding performers.

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Houston, Dallas, Greggton, Pampa and Odessa, Texas Tulsa Shreveport St. Louis Los Angeles

Steel plates and bolts reinforce gate valves.

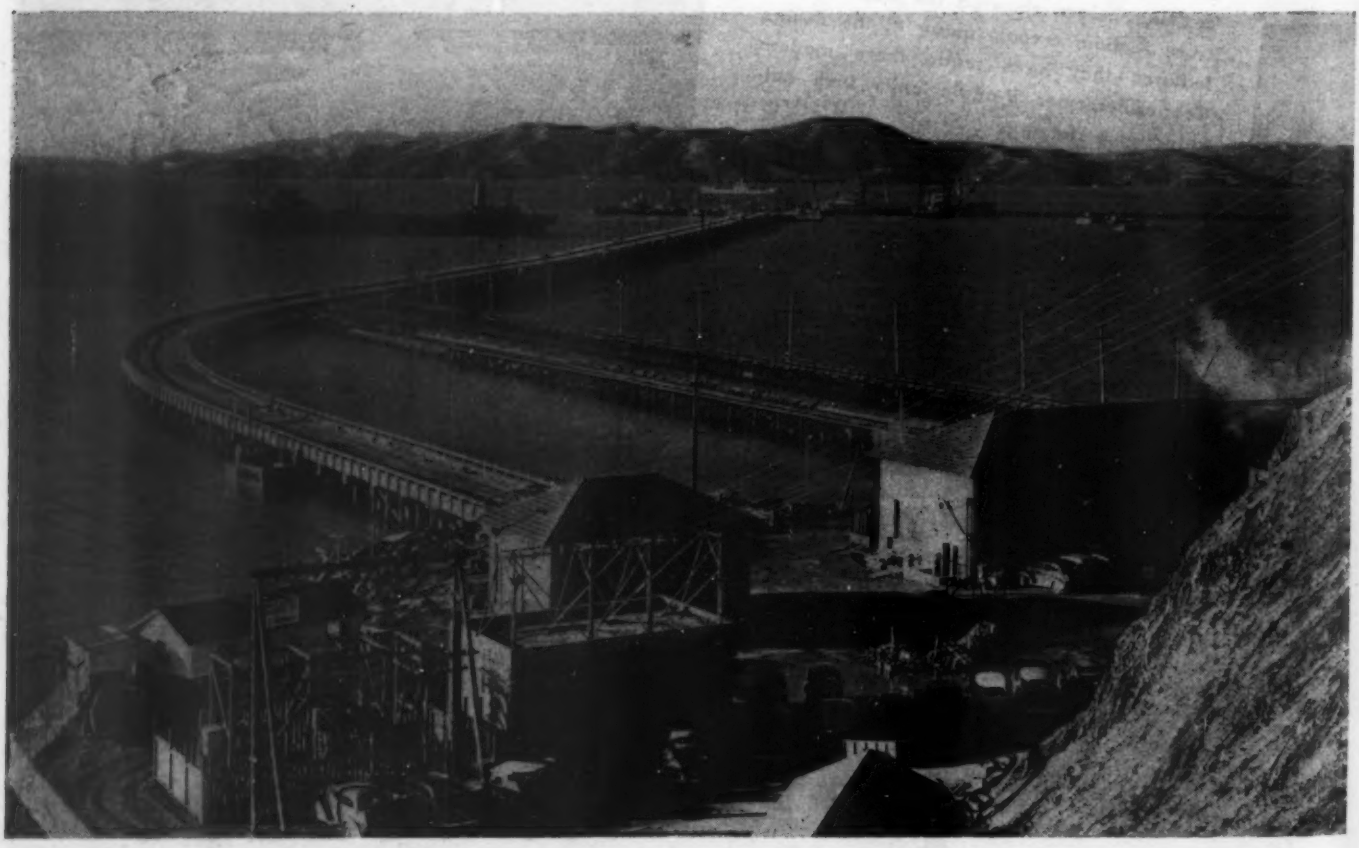
ditions. To prevent damage to large low-pressure tank valves, either from pipeline thermal expansion, earthquake stresses, or other cause, Standard Oil Company of California reinforces these valves with heavy steel plates. Two square plates of desired thickness are used for each valve. They are torch-cut to suitable bore and bolt circle to match the valve and pipe flanges. Four holes are provided to accommodate tie bolts which are used to clamp the plates against the valve companion flanges. The tie bolts have two nuts at each end so that the plates are clamped against movement in either direction, and the valve body accordingly is to a large degree relieved of stresses resulting from either tension or compression loads.

Thread Protectors

ON FIRE equipment which must be in condition for instant use, Kendall Refining Company, Bradford, Pennsylvania, installs wood covers for the threads to which fire hose is connected. The covers are made of two plies of one inch planking nailed to supply reinforcing by cross-graining, and sawed with octagonal faces.

The wood is bored in the center for a tight fit over the threaded nipple. Threads are not run into the wood, so that a quick pull easily removes it, yet it will remain in place indefinitely if not disturbed.

Ladders used where the ground is bare, or the surface covered with chat or gravel, can be prevented from slipping with a spade-type attachment on the base. A sheet of tank steel, cut to shape and bolted to the legs of the ladder, with a lip two inches below the legs to provide the required bite will prevent slipping when the ladder is in use.



Standard Oil Company of California's $4,370,000 program for modernization of its Richmond refinery "Long Wharf," is now well along toward completion as evidenced by this panoramic view. The mile-long reinforced-concrete causeway, shown curving off to the left, is completed and soon will carry vehicular traffic to the end of the loading wharf. This will permit abandonment of the existing narrow gauge electric railway. The loading section of the wharf, constructed at right angles to the causeway, is being lengthened from its present 1516 feet to 2466 feet. There will be four deep-water berths for bulk loading on the outer face and one on the inner face, as well as space for six barges.

THE MONTH IN THE INDUSTRY

API Midyear Meet At St. Louis June 2-3

The Division of Refining of the American Petroleum Institute will hold a midyear meeting in St. Louis June 2 and 3. Dr. Robert E. Wilson, chairman of the board of Standard Oil Company (Indiana) and American Petroleum Institute vice president for refining, said technology will be stressed in a program whose details will be made public shortly. An open dinner meeting is to be held the evening of June 2.

Activities of the meeting will be in the Jefferson Hotel.

During the war period midyear meetings of the Institute were dropped from the list of activities. Directors approved resumption of midyear programs by the several divisions with the programs to be worked out as conditions permit.

Refiners' San Antonio Program Arranged

Both synthetic fuel production and petro-chemical processing will make up parts of the program of the thirty-fifth annual meeting of the Western Petroleum Refiners Association, Plaza Hotel, San Antonio, March 24, 25 and 26.

The first day will be given over to registration and meetings of technical and finance committees. That evening the buffet supper with the compliments of Ethyl Corporation will mark the first entertainment feature of the program.

The first technical session the morning of March 25 will be presided over by L. D. Mann, Cities Service Refining Corporation. T. H. Risk, Ethyl Corporation, Detroit, will present a paper, "Motor Fuel Trends in Relation to Anti-Knock Requirements." This will be followed by a panel discussion on catalytic cracking. Discussion will follow each paper.

Governor Beauford Jester of Texas will give an address of welcome at the general session that afternoon, when R. L. Tollett, Cosden Petroleum Corporation, will preside. Following the opening remarks of H. T. Ashton, association president, the following technical subjects will be presented:

"Petroleum and Natural Gas—Uses and Possible Replacement," R. E. Wilson and J. K. Roberts, Standard Oil Company (Indiana).

Directors and officials will be elected at this session. Entertainment that evening will be provided by the supply and transportation companies which serve the refining industry.

A technical session the morning of March 26 will have these subjects:

"Petroleum Chemicals," M. B. Chittick, The Pure Oil Company.

"Synthetic Fuel Production by the Hydrocol Process," P. C. Keith, Hydrocarbon Research, Inc.

G. R. Kenter, Tidewater Associated Oil Company, will preside at this session. A meeting of the directors will be held at 10 o'clock.

The final general session will be held the afternoon of March 26 with the following subjects:

"Various Factors in the Future Supply and Demand Picture," S. A. Swensrud, Standard Oil Company (Ohio).

"Petroleum Industry Public Relations Program," Ralph C. Champlin, Ethyl Corporation.

R. W. McDowell, Mid-Continent Petroleum Corporation, will preside.

The annual dinner-dance will close the meeting the evening of March 26.

API Marketing Group Sponsors Meeting

Petroleum marketers, large and small, will gather at French Lick, Indiana, April 1-2 for a national oil marketing meeting sponsored by the General Marketing Committee of the American Petroleum Institute. P. E. Lakin, Shell Oil Company, New York, program chairman, announced that three nationally-known speakers will appear on the two-day program: James A. Doolittle, vice president of Shell Union Oil Corporation; Dr. Claude Robinson, president of Opinion Research Corporation, and Charles Roberts, Stewart-Dougall and Associates, marketing research consultants.

ASME Boiler Committee Plans Hearings on Code

The Boiler Code Committee of the American Society of Mechanical Engineers has made arrangements to hold two public hearings in the Southwest on proposed revision of the Unfired Pressure Vessel Code. The first of these hearings will be at the Rice Hotel, Houston, May 1 at 8 p.m. and May 2 at 9:30 a.m. The second will be at the Biltmore Hotel, Los Angeles, May 7 at 9:30 a.m. The purpose of these hearings is to give manufacturers and users of pressure vessels, and state enforcement authorities opportunity to verbally comment on the proposed revision. All those interested have been urged to attend.

A previous announcement by the Boiler Code Committee advised that its Special Committee to Revise Section VIII of the ASME Boiler Construction Code (Unfired Pressure Vessel Code) has submitted the Proposed Revision of Section VIII on the code in draft form, dated January, 1947.

For those interested in reviewing the proposed revision, copies are obtainable from the ASME at 29 West 39th Street, New York 18, N. Y., at $1.00 per copy. All those interested have been invited to submit their comments in writing to the secretary of the Boiler Code Committee, whether or not they are able to attend one of the hearings.

The Houston hearing will be under the sponsorship of the ASME South Texas Section, and the one at Los Angeles under that of the Southern California Section. Members of the Boiler Code Committee and its subcommittes are planning to attend these hearings. The Boiler Code Committee will hold its May meeting at the Biltmore Hotel, Los Angeles, May 9.

The National Board of Boiler and Pressure Vessel Inspectors also is holding its general meeting on May 5, 6 and 8 at the Biltmore Hotel in Los Angeles.

Conventions

March

17-19—American Society of Lubrication Engineers, Hotel William Penn, Pittsburgh.
24-26—Western Petroleum Refiners Association, annual membership meeting, San Antonio, Texas.
24-30—Oil Heat Institute of America, Traymore Hotel, Atlantic City, New Jersey.

April

7-10—National Association of Corrosion Engineers, Palmer House, Chicago.
17-18—National Petroleum Association, Cleveland Hotel, Cleveland.
23-25—Natural Gasoline Association of America, Baker Hotel, Dallas.
30-2—Natural Gas Department, American Gas Association, Stevens Hotel, Chicago.

May

4-7—Petroleum Equipment Suppliers Association, Mark Hopkins Hotel, San Francisco.
6-8—Petroleum Industry Electrical Association and Petroleum Electrical Supply Association, Rice Hotel, Houston.
6-8—Southwest Gas Measurement Short Course, University of Oklahoma, Norman.

June

2-3—American Petroleum Institute, Division of Refining, Midyear Meeting, Jefferson Hotel, St. Louis.
23-25—American Chemical Society, Midwest Regional Meeting, Kansas City, Missouri.

September

15-17—National Butane-Propane Association, Jefferson Hotel, St. Louis.
29-3—American Gas Association, San Francisco.

October

21-25—Pacific Chemical Exposition, San Francisco.

November

10-13—American Petroleum Institute, Stevens Hotel, Chicago.

California Refiners' Association Re-Elects

C. A. Johnson, Socal Oil & Refining Company, was re-elected president of the Independent Refiners' Assocation of California at a meeting of directors on February 11.

D. B. O'Neill, vice president and general manager, and M. M. McCallen, McCallen Refining Company, vice president, also were re-elected.

W. G. Krieger, of Krieger Oil Company, and J. D. Sterling, Eagle Oil & Refining Company, were elected to the board of directors. The following board members were re-elected: B. E. Devere, Pathfinder Petroleum Company; D. S. Fletcher, Fletcher Oil Company; Clifford Hancock, C. A. Johnson and M. M. McCallen.

Pitzer Speaks Before ACS Southeast Texas Section

The Southeastern Texas Section of the American Chemical Society met at Baytown February 13 to hear Kenneth Pitzer of the University of California talk on "Relationship Between the Mo-

lecular Structure of Hydrocarbons and Their Thermodynamic Properties."

The technical session was preceded by an inspection tour arranged by Humble Oil & Refining Company through portions of the laboratory and processing facilities of Baytown refinery, and by a group dinner.

The meeting was conducted by Briggs Manuel, chairman of the section, and the speaker was introduced by Sherman Schafer, chairman-elect. Dr. Pitzer presented slides showing results of experimental work and calculations on the energy-structure relation of hydrocarbons varying from light gases well into "gasoline-boiling-range" materials, and commented briefly on proposed experimental work along the same lines.

NPA Semi-Annual Meeting Announced

The forty-fourth semi-annual meeting of the National Petroleum Association will be held in Cleveland April 17 and 18. Sessions will be in the Cleveland Hotel.

Process Control Discussed At February CNGA Meeting

The successful application of automatic process controls involves the solution of three separate problems, E. H. Reynolds, Richfield Oil Corporation, told California Natural Gasoline Association members at their February 6 meeting where he presented a paper in which Wm. Troutman and Gordon Lawn, also of Richfield Oil Corporation, collaborated. These problems, Reynolds stated, are the rapid and accurate measurement of the variables of the process, rapid and precise application of corrective measures to maintain the controlled variables at the desired points with minimum deviation, and proper selection of the variables to be controlled.

The first two problems can be solved by the proper selection of control instruments, Reynolds said, and described the various types available. Asking that these be kept in mind, he then proceeded to discuss the third problem. The solution of this, he said, requires a study of the process and consideration of the products, or results, desired. For illustration, he presented diagrams of four typical processes and discussed for each the factors influencing the choice of variables to be controlled, the location of their points of measurement, and the selection of control instruments.

Military Aircraft Fuels Committee Appointed

Appointment of a temporary committee on military aircraft fuels productive capacity, authorized at the January meeting of the National Petroleum Council in response to a request of the government's Oil and Gas Division for a study of the problem of obtaining an authoritative technical evaluation of refining capacity for production of fuels for military aircraft, has been announced by Chairman Walter S. Hallanan.

Bruce K. Brown, Standard Oil Company (Indiana), was named as chairman of the committee, which will include R. C. Alden, Phillips Petroleum Company; Paul G. Blazer, Ashland Oil & Refining Company; Reid Brazell, Leonard Refineries, Inc.; George Davidson, Standard Oil Company of California; C. E. Davis, Shell Oil Company; A. P. Frame, Petroleum Advisers, Inc.; W. M. Holaday, Socony-Vacuum Oil Company; Arthur E. Pew, Sun Oil Company; G. H. Taber, Sinclair Refining Company; H. D. Wilde, Humble Oil & Refining Company, and J. A. Worden, The Texas Company.

New ACS Officers Elected At Lake Charles

The Southwest Louisiana Section of the American Chemical Society recently held its first election since organization last December, according to a report received from A. R. Rescorla, Cities Service Refining Corporation, Lake Charles, who has served as acting-chairman of the new section. New officers, who will serve during 1947, include A. R. Rescorla, chairman; W. A. Wilson, vice-chairman; T. W. Kirby, secretary; R. W. Rice, treasurer, and E. G. Anderson, councilor. Activities of the section now include publication of a monthly "Newsletter for technical personnel" which is distributed to the membership, and which will carry some technical material in addition to news of local doings.

Western Petroleum Refiners Adds Public Relations

Expansion of the activities of Western Petroleum Refiners Association to include industrial relations has been announced by H. T. Ashton, president. James T. Rasbury has been named manager of the new department, which will keep the membership and the industry advised on current legislative matters, decisions, rules and regulations and in addition, all statistical information considered important and beneficial.

Rasbury is a graduate of Michigan State Normal College. Later he completed graduate work at the University of Oklahoma and at Washington University. Recently he was admitted to the bar of the District of Columbia. His industrial experience includes connections with Studebaker Corporation and the Federal Bureau of Investigation.

New CNGA Chapter Formed

A California Natural Gasoline Association chapter has been formed at Ventura, and will be known as the Coastal Chapter. C. L. Case, Continental Oil Company, is chairman, and G. C. Elmore, Tide Water Associated Oil Company, is secretary. Meetings are to be held on the third Wednesday of each month. The initial meeting in February had 85 in attendance.

Views of FPC Staff, Given in Report Coincide With Industry on Regulation

The Federal Power Commission is in agreement with the gas and oil industries that the production, gathering or processing of natural gas by persons who do not move it beyond the point where the gathering function has been completed but sell the gas "at arm's length" to others who transport and sell it in interstate commerce, is exempt from FPC jurisdiction under the provisions of the Natural Gas Act.

This position was taken in a staff report on the subject, being distributed to the industry for comment before being incorporated in the report which the commission will send to Congress soon.

"No reasonable basis is found in the act or its legislative history for an interpretation that, although the 'activities' of production and gathering are exempt under Section 1 (B), sales of natural gas which are made at arm's length by producers and gatherers who do not thereafter transport it in interstate commerce may be regulated," it was declared. "Unless such a distinction is specifically disclaimed, doubts and apprehensions will continue to be felt and expressed regarding the possible application of the natural gas act to the operations of those who only produce and gather natural gas."

Control of Oil Involved

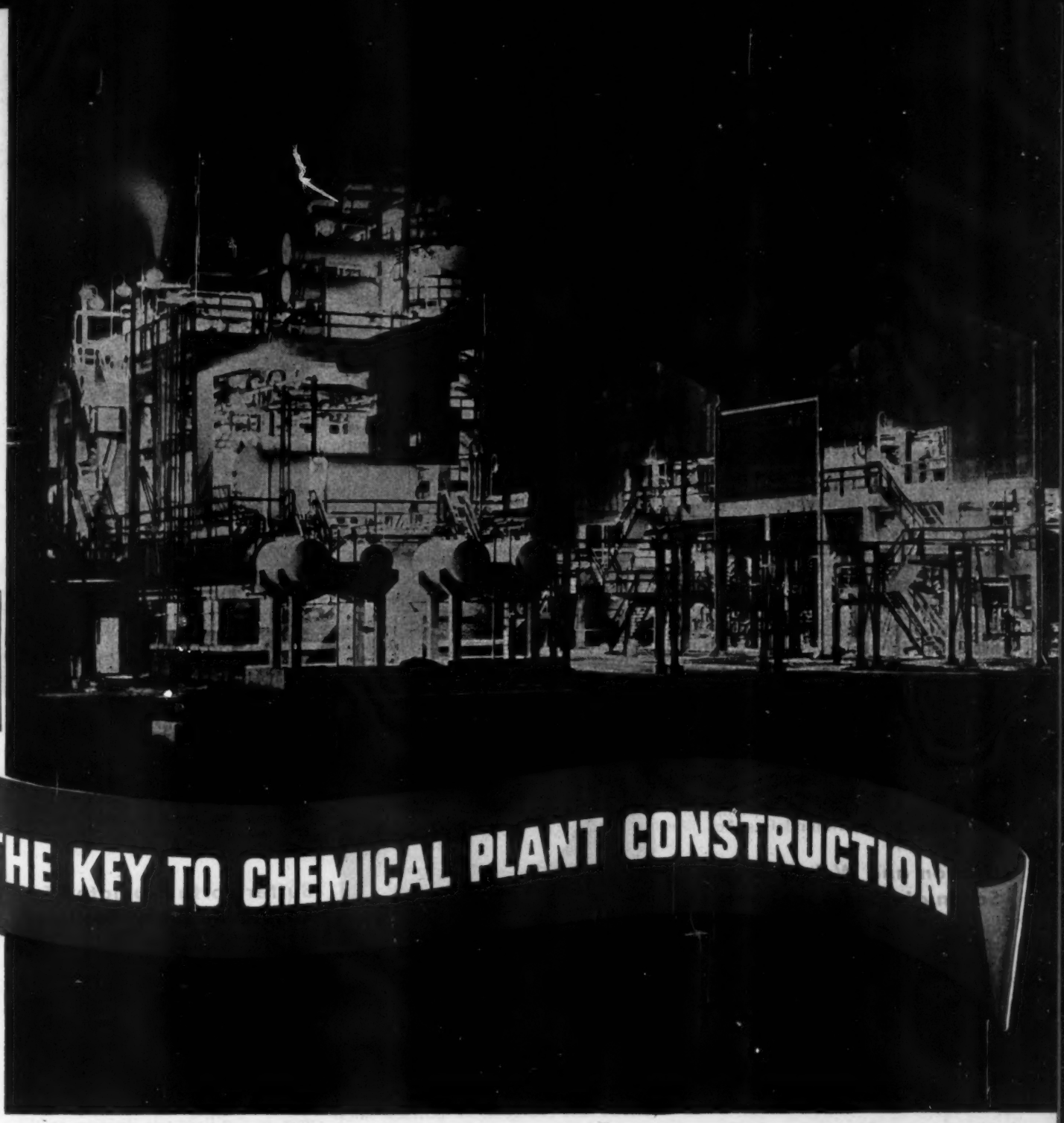
The report pointed out that if FPC or any other federal agency were to be authorized to control arm's length sales by producers and gatherers, the result would be to establish federal authority over all field and well prices for natural gas and "control of oil as well as gas would necessarily become involved, since the production of these two resources is to a large extent inseparable."

It would not be practicable for the government to attempt to regulate sales of natural gas by producers and gatherers unless it also took over from the states essential functions now performed by them relative to conservation and the protection of correlative property rights, it was added.

"In view of the widespread uncertainty which has been manifested regarding the commission's view of its jurisdiction over production and gathering sales, it is important that the commission take appropriate action to relieve the dobuts and fears now prevailing in tihs regard," the staff cautioned.

This could be done either by formal adoption of an administrative rule or by recommendation of an amendment to Section 1 (B), although it is felt that the latter is not necessary. However, whichever procedure is followed, it was declared, it should be made clear and certain that these activities, when not connected with interstate commerce, are exempt from FPC jurisdiction.

The discussion of production was one of four sections of the report, in another of which state conservation laws and activities were discussed and the recommendation made that the producing states which have a flare gas problem consider the enactment of legislation prohibiting flaring of casinghead gas "except as specifically and temporarily authorized by state commission order." A limited period of grace would be desirable before such prohibition became effective, so producers could adjust their operations, it was said, and it might be necessary also to extend sole discretion to the conservation authorities to grant temporary exemptions for new wells and



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small allowances for flaring in remote areas.

The report took the firm position that the conservation of gas is a responsibility of the states, but a responsibility which must be met.

The other sections of the report dealt, respectively, with practices and problems in producing, gathering and processing natural gas, and with gas reserves of the nation, in which it accepted DeGolyer's estimate of reserves of slightly more than 144 trillion cubic feet as of January 1, 1946, as the basis of a discussion of location and ownership of known reserves and the prospects for future discoveries.

Exploratory Costs Increasing

Experience suggests that the future prospects for continued substantial additions to the reserves of both gas and oil are generally factorable, it was concluded, but the experience of the oil industry shows that costs of exploration and discovery are increasing rapidly and "prospects for future discovery of oil are not as encouraging as would appear to be indicated by the periodic estimates of year-end reserves," and oil and probably gas may be more difficult to find in the future. In deep drilling, however, the chances of discovering oil rather than gas are increasing and on the whole, it was said, "it would be reasonable to expect that many new additional discoveries of gas will be made before a decline sets in."

The reports were released as the House Interstate and Foreign Commerce Committee announced two days of hearings beginning March 18 on bills for amendment of the Natural Gas Act.

Gibbon and Wright Given Socony Indies Appointments

Harry A. Gibbon has been appointed general manager in the Netherlands Indies for Nederlandsche Kolonial Petroleum Maatschappij, with Albert Wright as his assistant. N. K. P. M., subsidiary of Standard-Vacuum Oil Company, has a large refining installation near Palembang and oil fields in Sumatra.

Gibbon is a graduate of The Rice Institute, and after working with Humble Oil & Refining Company in Texas, joined Standard-Vacuum in 1939. He was transferred to N. K. P. M. after the war and proceeded to Batavia, Java, after the Japanese surrender made it possible for that company to start planning rehabilitation of its refinery and oil-field properties in Sumatra.

Wright was graduated with a degree in geology from Harvard. He has specialized in production work and joined N. K. P. M. in 1938 after 14 years experience in the oil fields of Venezuela and the Middle East. He has been production manager, and recently has been assigned to the company's headquarters at the Hague.

Jefferson Chemical Announces Appointments

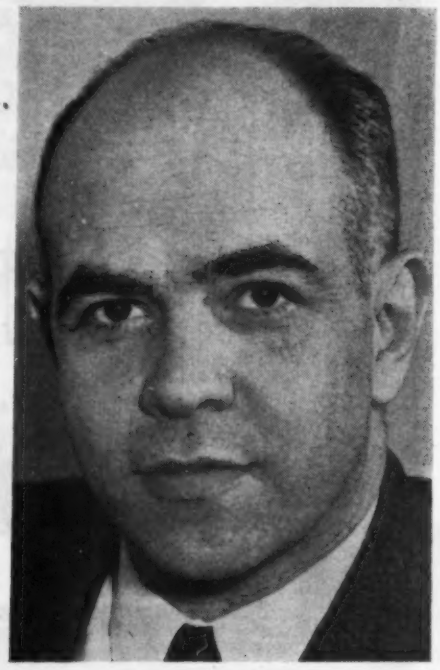
Dr. Max Neuhaus, formerly director of research for Jefferson Chemical Company, has been named manager of the technical and research department, with

DR. MAX NEUHAUS

Dr. Paul P. McClellan and John C. Paul as assistant managers.

Dr. Wm. H. Bowman, formerly market development manager, has been designated sales department manager, and G. W. Larson has joined the department's staff to take charge of its technical sales service work. Larson was until recently with the Barrett Division of Allied Chemical and Dye Corporation.

Peter Harrison of the engineering department has been named purchasing agent, and G. R. Wick has been named assistant chief engineer.



DR. W. H. BOWMAN

Jefferson Chemical Company's plant at Port Neches, Texas, is scheduled to begin production during the latter half of this year. Its initial products will include ethylene oxide and ethylene glycol. The company's executive offices are at 30 Rockefeller Plaza, New York.

Butterworth Promoted To Casper Refinery Position

L. H. Butterworth, assistant general foreman in the light-oils division of the Whiting refinery of Standard Oil Company (Indiana), has been promoted to assistant general superintendent of the company's Casper, Wyoming, refinery.

Butterworth joined the organization in 1937 as a chemical engineer in the research department at Whiting. In 1942 he was transferred to the manufacturing department as an assistant general foreman in the light-oils division at Whiting, serving first in the light-oils finishing department and later in the crude stills department. He received his bachelor of science in chemical engineering from the University of Iowa.

Voorhies Named Director of Esso Lab at Baton Rouge

Alex Voorhies, Jr., has been named director of Esso Laboratories in Baton Rouge to succeed Dr. C. L. Brown, who has become a member of the research and development coordination group of Standard Oil Development Company, New York. Other changes at the Baton Rouge laboratory are the promotion of Dr. R. W. Richardson to associate director, and Dr. R. W. Krebs to assistant director.

The new director has been at the Baton Rouge laboratory since 1930,

Trends of Operations and Changes in Stocks

Figures on crude stocks are from Bureau of Mines weekly reports; all others from American Petroleum Institute weekly reports, which are estimates on Bureau of Mines basis.

(All figures in thousands of barrels—add 000)

Trends in Week Ended	Crude Oil			Gasoline		Gasoil and Distillate		Residual Fuel	
	Production Daily	Runs to Stills Daily	Stocks Week End	Production Weekly	Stocks Week End	Production Weekly	Stocks Week End	Production Weekly	Stocks Week End
1946:									
January 26	4,626	4,553	220,544	13,622	101,737	5,720	29,498	8,411	39,722
March 2	4,726	4,779	229,430	13,871	104,462	5,888	25,148	8,634	38,441
March 30	4,425	4,684	224,994	13,896	104,715	5,337	28,240	8,738	37,746
April 27	4,672	4,685	224,443	14,228	99,631	5,568	30,466	9,204	39,404
May 25	4,759	4,857	222,214	14,322	95,769	5,463	32,973	8,908	43,368
June 29	4,957	4,854	223,883	14,500	92,333	5,325	37,762	8,828	46,447
July 27	4,926	4,896	223,756	14,535	88,626	5,817	44,316	8,217	49,517
August 24	4,836	4,866	225,672	14,639	86,251	5,649	51,405	8,126	52,061
September 28	4,778	4,829	221,903	14,538	85,854	5,450	57,903	8,158	56,914
October 26	4,730	4,758	221,184	14,863	86,423	5,710	65,499	7,728	60,872
November 30	4,795	4,707	225,119	15,145	88,371	5,228	66,062	7,672	58,647
December 28	4,713	4,968	226,111	15,604	93,126	5,931	58,941	8,181	53,427
1947:									
January 25	4,672	4,820	221,655	14,624	99,801	5,630	50,357	8,224	48,558
February 22	4,786	4,860	224,580	14,668	103,904	5,929	40,739	8,542	44,919

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Brands of STABILIZED SULFURIC ANHYDRIDE

General Chemical Company—America's pioneer with the Contact Process for manufacture of high strength sulfuric acid and oleum—makes another major contribution to Basic Chemicals for American Industry with SULFAN . . . Anhydride of Sulfuric Acid.

By perfecting new methods of stabilizing Sulfur Trioxide, General Chemical Research takes an invaluable chemical tool off the shelf of laboratory curiosities and brings it to the Process Industries as a chemical of commerce for use in a host of ways.

General Chemical offers SULFAN in three chemically equivalent forms:

Sulfan 'A' Partially Stabilized, melting at approx. 35°C;

Sulfan 'B' Completely Stabilized, melting at approx. 17°C; and

Sulfan 'C' Unstabilized.

Experimental samples and further technical information are available on request from General Chemical Company, Research and Development Division, 40 Rector Street, New York 6, N. Y.

Some Potential Uses

1. For fortification of spent oleum, making possible a ready supply of any strength oleum.
2. In benzenoid sulfonations for elimination of mixed sulfonates: meta- only or ortho- and para- derivatives only are formed.
3. For di- and poly-sulfonations of aryl compounds.
4. For direct sulfonation of aliphatics.
5. For sulfonations in the presence of a solvent, thus eliminating the removal of H_2SO_4 necessary when oleum is the agent.
6. For formation of addition compounds with amines, valuable in organic synthesis.

Sulfuric Anhydride exists in three chemically equivalent physical modifications as indicated by data below. General Chemical's stabilized product is almost entirely Gamma-Form and its partially stabilized product is largely Beta-Form.

PROPERTY	GAMMA-FORM	BETA-FORM	ALPHA-FORM
Description	Ice-Like	Asbestos-Like	Asbestos-Like
Equilibrium Melting Point (°C):	16.8	32.5	62.3
Density (20°C)	1.9255	—	—
Sp. Ht. (cals/gm) (20°C)	0.77	—	—
Ht. of Fusion (cals/mol)	1,800	2,900	6,200
Ht. of Sublimation (cals/mol)	11,900	13,000	16,300
Ht. of Dilution (cals/mol)	40,340	—	—
Vapor Pressure (mm.)			
0°C	45	32	5.8
25	433	344	73
50	950	950	650
75	3,000	3,000	3,000

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when he left the faculty of Loyola University, New Orleans. He is a graduate of Louisiana State University with a degree in chemical engineering. His master's work was done at Loyola.

Dr. Richardson, a graduate of Louisiana State University with graduate work at Iowa State University, began his work as a chemical engineer in the refinery in 1933. From 1937 to 1940 he was at the Esso Laboratories in New Jersey.

Dr. Krebs is a graduate of the University of Illinois, where he was on the faculty before joining the laboratory staff 10 years ago.

J. Howard Pew Steps Down as Sun President

Along with the announcement of his resignation as president of Sun Oil Compay, J. Howard Pew recommended election of Robert G. Dunlop to the post. Further he recommended to the board of directors that Joseph N. Pew, Jr., now executive vice president, be named chairman of the board, a new position. The resignation of Arthur E. Pew, Jr., vice president in charge of manufacturing, was announced.

It also was recommended that the board of directors be increased to 13 from 11 and that an executive committee be named. Pew said he would continue as a director, and that Arthur E. Pew, Jr., also would continue as a director.

Robert G. Dunlop joined Sun Oil Company in 1933, after two years in private accounting work. In 1941 he was appointed assistant comptroller and in 1944 he was named comptroller and elected to the board of directors.

Texas City Plant to Resume Operations

Resumption of operations of the Texas City plant of Sid Richardson Refining Company is slated for April 1. Freeman W. Burford has been elected president of the company, which was organized following purchase of the plant from American Liberty Oil Company.

During the war the plant was a source of 100-octane gasoline. Since then its operation has been spasmodic. Alterations were begun after formation of the new company, which took possession March 1. Further expansion of facilities is under consideration.

New Company Plans Wax Plant in Houston Area

Paraphalt Wax Corporation of America, a new organization, has announced plans for a paraffin wax-asphaltum plant to be erected in the Houston area. Construction is proposed to be completed this year. Dr. Ernest Stossel is to be president of the new organization, and A. T. Gohlson, vice president. Dr. Frank Jessen, Associated Engineers of Houston, will be associated with the organization, Gohlson is vice president of Hovey Petroleum Company.

Dr. Stossel is head of several similar organizations in South America. He is a graduate of Vienna University where he specialized on oxidation of hydrocarbons. He worked in Spain in the field of vegetable oils and soaps.

API-AGA Joint Estimates Show Oil and Gas Reserve Increase

The continental United States has proved liquid hydrocarbon reserves of 24,194,587,000 barrels and proved natural gas reserves of 160.6 trillion cubic feet, as of December 31, 1946.

These figures were disclosed in a joint report of the Committees on Reserves of the American Petroleum Institute and the American Gas Association.

Of the total liquid hydrocarbon reserves, 20,873,560,000 barrels are crude oil and 3,321,027,000 barrels are natural gas liquids. Similar figures for the year previous are not available, but the table shows that crude oil reserves increased 931,714,000 barrels, or approximately 5 percent as follows (in barrels):

	Dec. 31, 1945	Dec. 31, 1946
API crude oil reserves	19,941,846,000	20,873,560,000
API condensate reserves	884,967,000	1,050,794,000
Additional natural gas liquids reserves	(not available)	2,270,233,000
Total reported liquid hydrocarbon reserves	(not available)	24,194,587,000

(Natural gas liquids include condensate, natural gasoline and liquefied petroleum gases. Where condensate is produced and run with crude oil, it is treated as crude oil in these estimates.)

Proved natural gas reserves of 160.6 trillion cubic feet increased 12.8 trillion cubic feet from the December 31, 1945, total of 147.8 trillion, or about 8 percent. The estimated net production of natural gas for the year of 1946 is 4.9 trillion cubic feet after deducting the amount of gas returned to reservoirs.

Heretofore API estimated U. S. reserves of liquid hydrocarbons which included all of the crude oil and the condensate portions of the natural gas liquids. This year, API and AGA divided the work into crude oil reserves, estimated by the institute, and natural gas reserves with their contained liquids, estimated principally by AGA, assisted by API.

The API Committee on Reserves as of December 31, 1946, estimated that the proved reserves of crude oil in the U. S., exclusive of condensate, was as follows (barrels of 42 U. S. gallons):

Total proved reserves of crude oil (exclusive of 884,967,000 barrels of condensate) as of December 31, 1945		19,941,846,000
Revisions of previous estimates	+1,254,705,000	
Extensions of old pools	1,158,923,000	
New reserves (new pools) discovered in 1946	244,434,000	2,658,062,000
Total proved reserves as of December 31, 1945, and new proved reserves added in 1946		22,599,908,000
Subtract production during 1946		1,726,348,000
Total proved reserves of crude oil as of December 31, 1946		20,873,560,000
Increase in crude reserves since December 31, 1945		931,714,000

Estimates in the report, as in all such previous annual reports, refer solely to proved or blocked-out reserves and include only oil recoverable under existing economic and operating conditions.

"For proved reserves as of December 31, 1944, we showed 668,701,000 barrels condensate and 19,784,530,000 barrels crude oil, a total of 20,453,231,000," the report says. "As of December 31, 1945, we showed 884,967,000 barrels condensate and 19,941,846,000 barrels crude oil, a total of 20,826,813,000. Analagous figures for December 31, 1946, are 1,050,794,000 barrels condensate and 20,873,560,000 barrels crude oil, a total of 21,924,354,000. In other words, 1,050,794,000 barrels condensate for December 31, 1946, is the AGA Committee's present estimate of condensate reserves in the

Estimated Proved Reserves in the United States

(Thousands of Barrels of 42 U. S. Gallons.)

STATES	Estimated Proved Reserves Crude Oil Plus Condensate (Dec. 31, 1945) as shown in Committee's Report of February, 1946 (1)	PROVED RESERVES OF CRUDE OIL—(Condensate not Included) Proved Reserves as of Dec. 31, 1945 (2)	Changes in Proved Reserves Due to Extensions (New Oil) and Revisions in 1946 (3)	Proved Reserves in New Pools Discovered in 1946* (4)	Production During 1946 (5)	Proved Reserves as of Dec. 31, 1946 (Columns 2+3+4 Less Column 5) (6)
Alabama	785	785	109	.....	382	512
Arkansas	303,674	287,623	4,172	1,764	26,501	267,058
California	3,409,948	3,318,006	276,787	13,876	315,178	3,293,491
Colorado	259,830	259,830	51,265	800	12,025	299,870
Illinois	349,620	349,620	72,452	4,606	75,414	351,264
Indiana	41,243	41,243	7,878	1,633	6,684	44,070
Kansas	541,846	541,846	87,319	12,629	96,478	545,316
Kentucky	56,721	56,721	13,066	88	10,687	59,188
Louisiana	1,689,781	1,558,891	207,018	25,857	139,969	1,651,797
Michigan	64,186	64,186	21,576	415	17,000	69,177
Mississippi	267,160	257,363	34,939	1,790	23,871	270,221
Montana	108,474	108,474	2,576	2,000	8,804	104,246
Nebraska	500	500	665	.....	290	875
New Mexico	512,373	512,373	65,000	2,871	36,791	543,453
New York	81,189	81,189	.....	.....	4,900	76,289
Ohio	29,681	29,681	3,047	.....	3,508	29,220
Oklahoma	889,839	888,839	98,423	47,731	136,807	898,186
Pennsylvania	110,601	110,601	.....	.....	13,100	97,501
Texas	11,470,294	10,835,257	1,441,158	126,594	756,649	11,646,360
West Virginia	38,630	38,630	.....	.....	2,975	35,655
Wyoming	600,131	599,881	25,919	1,780	38,222	589,358
Miscellaneous†	307	307	259	.....	113	453
Total U.S.	20,826,813	19,941,846	2,413,628	244,434	1,726,348	20,873,560

* Only a limited area is assigned to each new discovery even though the committee may believe that eventually a much larger area will produce, for in this report the concern is only with actually proved reserves.

† Includes Florida, Missouri, Tennessee, Utah and Virginia.

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Condenser and boiler feed water plant of Dallas Power & Light Company's Mountain Creek Plant.

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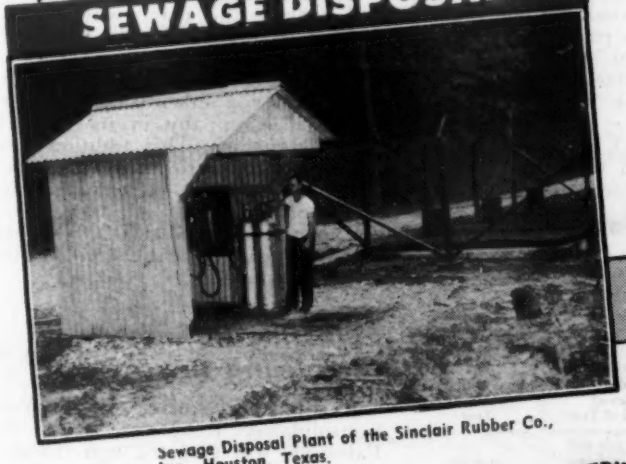
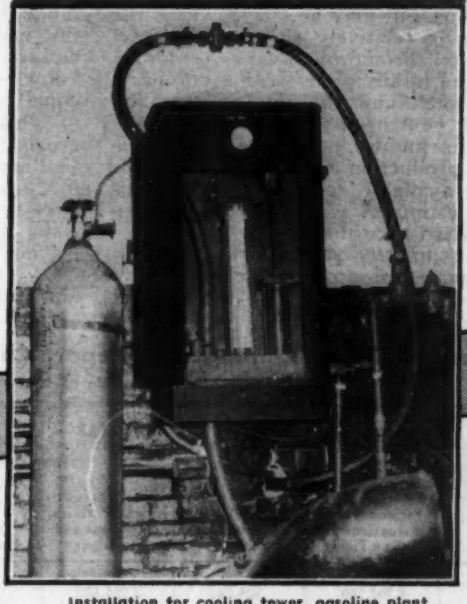


Sewage Disposal Plant of the Sinclair Rubber Co., Inc., Houston, Texas.

ALGAE CONTROL



Installation for cooling tower, gasoline plant near Vivian, La.

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same fields which were included in previous years by the API Committee. If comparison is made between new oil reported in 1946 with new oil reported in earlier years, it should be remembered that the 1946 figures for new oil do not include new condensate estimated at about 205,000,000 barrels.

"The estimates for this report do **not** include:

1) Oil under the unproved portions of partly developed fields;

2) Oil in untested prospects;

3) Oil that may be present in unknown prospects in regions believed to be generally favorable;

4) Oil that may become available by secondary-recovery methods from fields where such methods have not yet been applied;

5) Oil that may become available through chemical processing or natural gas; or

6) Oil that can be made from oil shale, coal, or other substitute sources.

"Proved reserves include 1) *Proved drilled reserves* or oil estimated to be recoverable by the production systems now in operation, (primary or secondary) and from the area actually drilled up on the spacing pattern in vogue in that pool; and 2) *proved undrilled reserves,* or oil under undrilled spacing units which are so close, and so related, to the drilled units that there is every reasonable probability that they will produce when drilled.

"In the case of new discoveries, the estimates of proved reserves necessarily represent but a part of the reserves which may ultimately be assigned to the new reservoirs. Where development has not yet gone beyond the discovery well, the area assigned as proved is usually small in regions of complex geological conditions, but may be larger where the geology is relatively simple. The total of new oil through discoveries estimated as proved in any given year is comparatively small and the total of new oil through extensions is comparatively large. As knowledge of the factors affecting production and well performance become available, reserves in older fields can be estimated with greater precision and revised accordingly."

The committee stressed the fact that its estimates of proved reserves cannot be used as a measure of the rate at which these reserves can be produced with or without physical waste. "Today's known oil can be recovered only over a period of many years and at gradually declining annual rates," it said. "Therefore, only incorrect conclusions as to the life of these estimated reserves can be obtained by dividing these reserves by the current rate of production."

The committee, headed this year by F. H. Lahee, Sun Oil Company, Dallas, emphasized that every effort was again made to get a fair, unprejudiced, and representative opinion. All previously determined factors pertaining to the various pools were examined and adjusted in the light of new information.

Beaumont Chemists Hear Army Chemical Chief

General Alden H. Waitt at Beaumont, February 19, addressed the Texas-Louisiana-Gulf section of the American Chemical Society, on problems of national defense as they appear from his viewpoint as Chief of the Chemical Corps, United States Army.

"The citizen," he said, "no longer can regard defense as something remote, something he can delegate to the military and then forget. Future war will come suddenly and intensively.

"Sabotage may be the major weapon at the start of the war. Enemy agents could infiltrate this country months and years ahead of attack, and it would be possible to bring piecemeal into the country super explosives which could be planted in critical places, timed to explode at the selected time. Fifth-column activities will be used to cause dissention and unrest among segments of the population, and, it is possible the fifth column may already have started. The enemy may no longer be dependent upon manually controlled aircraft for long-range bombardment. We must assume the enemy has developed the atomic weapon successfully, and that he has knowledge of toxic weapons at least equal to our own.

"This country has become the world's greatest power. I wonder if we realize that we will have to pay a heavy price if we are to maintain our position. We have to be equal in strength to any possible rival if we are to maintain our system.

"As Chief of the Chemical Corps, I am vitally concerned with the progress of chemical and allied sciences, and with the interest of the scientist, engineer, and producer in the defense program."

Plans for the Chemical Corps were described as emphasizing research and development; procurement planning and accumulation of reserve stocks; reserve training, and education of Regular Army officers.

"The Germans were reported to have developed a new toxic agent, termed 'tabun,' which is a highly effective weapon that fills a definite gap in usefulness between mustard gas and other toxic gases. Our thoughts will be geared to the discovery of chemical agents of many times the order of toxicity of those of World War I. Intense efforts must be made in the furtherance of this work, and second rate research is worse than none, as it leads to a false sense of security.

"Development work is also proceeding on incendiaries, flame weapons, and flame throwers, in which fields our services are reported to have excelled in the recent war. The postwar technical organization of the Chemical Corps is nearly complete. A research council has been organized of some 100 scientists. Manufacturing facilities for chemical warfare munitions will be maintained where possible. Plants which have peacetime application are available to industry on lease, which plan has the important advantage of maintaining these plants in operable condition."

General Waitt was introduced by C. O. Buttrill. V. A. Kalichevsky, chairman of the section, presided.

Two Lube Plants for Gulf Coast Refining Area

Contracts are being let for the buildings which will be a part of the new lubricating oil unit in the Smith's Bluff plant of The Pure Oil Company. The new facilities are calculated to cost $9,000,000, and approval of the Civilian Production Administration has been given for the buildings.

Lubricating-oil facilities at this plant will replace the plant which was destroyed by fire at Marcus Hook, Pennsylvania, prior to the war.

Approval of APA also has been announced for a lubricating oil unit for Shell Oil Company in its Deer Park plant (Houston). This addition was announced last year but plans were not pressed during the extreme shortage of building material.

Expenditure of $10,000,000 is contemplated for the lubricating-oil facilities of the Shell plant.

Palestine Refinery Increasing Capacity

Expansion of capacity of the plant of Consolidated Refineries, Ltd., Haifa, Palestine, will give the unit throughput of 100,000 barrels daily. Present rated capacity is around 80,000. Included in the expansion program are units for lubricating-oil manufacture.

The plant, owned by Anglo-Iranian Oil Company and Anglo-Saxon Petroleum Company, began operation in 1939. Since then, capacity has been increased by frequent additions.

Additional crude for the enlarged plant will be available when capacity of pipe lines to Near East production is put in operation.

Summary of API Committee's Report Covering Period 1937-1946

(Thousands of Barrels of 42 U. S. Gallons.)

YEAR	NEW OIL BLOCKED OUT DURING YEAR: Through Revisions to Previous Estimates and Extensions to Known Fields	NEW OIL BLOCKED OUT DURING YEAR: Through New Pools Discovered During Year	NEW OIL BLOCKED OUT DURING YEAR: Total Through New Discoveries, Extensions and Revisions	Production During Year	Estimated Proved Reserves as of End of Year	Increase Over Previous Year
1936	.....	.....	.....	.....	13,063,400	.....
1937	2,792,790	928,742	3,721,532	1,277,664	15,507,268	2,443,868
1938	2,243,571	810,493	3,054,064	1,213,186	17,348,146	1,840,878
1939	2,058,455	340,667	2,399,122	1,264,256	18,483,012	1,134,866
1940	1,607,012	286,338	1,893,350	1,351,847	19,024,515	541,503
1941	1,538,989	429,974	1,968,963	1,404,182	19,589,296	564,781
1942	1,618,925	260,051	1,878,976	1,385,479	20,082,793	493,497
1943	1,202,368	282,418	1,484,786	1,503,427	20,064,152	(—)18,641
1944	1,556,192	511,308	2,067,500	1,678,421	20,453,231	389,079
1945	1,690,315	419,984	2,110,299	1,736,717	20,826,813	373,582
1946	2,413,628	244,434	2,658,062	1,726,348	20,873,560	931,714

NOTE:—Up to and including its figures on proved reserves of petroleum as of December 31, 1945, the committee combined under that heading the estimated proved reserves of cycle plant and lease condensate. As of December 31, 1945 the reserves so included totaled 884,967,000 barrels, (leaving 19,941,846,000 barrels of crude oil only) and as of December 31, 1944 there were 668,701,000 barrels included. Beginning with this report the figures in this table are for crude oil only. If comparison is made between new oil reported in 1946 with new oil reported in earlier years, it should be remembered that the 1946 figures for new oil do not include new condensate which is estimated to be approximately 205,000,000 barrels.

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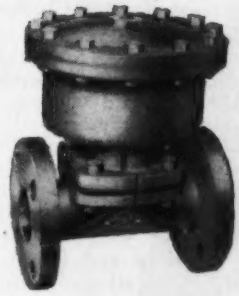

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Case History

Frequently the editorial staff of PETROLEUM REFINER meets this question, "How do you get articles for publication?"

An answer is at hand in *"Factors Affecting the API Design of Separators for Removal of Oil From Waste-Water Effluents."* In his letter of transmissal Sheppard Powell, consulting engineer of Baltimore, explained:

"Mr. W. B. Hart of The Atlantic Refining Company has suggested that we write you concerning publication of the enclosed paper.

"It has been our experience that most of our clients who have had reason to study the recommendations of the API manual, *'Disposal of Refinery Wastes, Section 1 Waste Water Containing Oil'* have been confused by the manner of presentation of data. In more than one instance we have observed as many answers in calculating separator design capacities as there were persons doing the calculating.

"As a result of this confusion we have reviewed some of the original experimental work which was made available through the courtesy of Mr. Hart . . . We have prepared this paper in order to call attention to certain of these apparent discrepancies. In our discussions with Mr. Hart he believed the paper should be offered to you for your consideration first, because of the subject matters."

One way to get manuscripts is to have the good fortune to be recommended, for which we thank Mr. Hart as well as Mr. Powell and his associate, E. L. Knoedler.

The article will appear shortly.

The April Issue

Last November during the annual meeting of the American Petroleum Institute the most popular of the technical papers seemed to be one of great length, "Miscellaneous Fractionating Techniques." Since then there have been requests for copies, which were available in the press room in Chicago.

Its length presented a problem, another of which was its preparation for publication. Scarcely a week since November has passed without some work being done in preparing this paper for presentation in the April, 1947, issue of PETROLEUM REFINER. Now it is down to the straight type setting and all that remains ahead of actual printing is proof reading by one of the six authors:

J. J. Cicalese and G. S. Houghland, The M. W. Kellogg Company; J. A. Davies, The Texas Company; P. J. Harrington, Standard Oil Development Company; A. J. L. Hutchinson, Fish Engineering Company; and T. J. Walsh, The Standard Oil Company (Ohio).

This manuscript really amounts to a textbook on fractionation, and its presentation in one issue of PETROLEUM REFINER will take care of the regrets that have come through failure to get preprints at the last API meeting.

Another shift in editorial content for the April issue, which will stress the processing involved in natural gasoline and cycling operations, came with arrival of a release through the engineering department of The Fluor Corporation "Evaluating Cooling Tower Performance for the Process Man," which will be a part of the April number.

Each year April draws its material from cycling and natural gasoline for the reason that the annual meeting of the Natural Gasoline Association of America is held then. This year it will be in the Baker Hotel, Dallas, April 23, 24 and 25.

Descriptions of some of the recent new cycling plants, an article on recovery of liquids by refrigeration in transmission, one on safety and the final chapter of the series on Basic Petroleum Chemistry by J. J. Stadtherr will be in this April issue.

NGAA Meeting

Getting all the subjects in the hands of proper and willing speakers for the annual meeting of the Natural Gasoline Association of America was the job given H. W. Harts, vice president, Warren Petroleum Company. The meeting will be held in Dallas April 23, 24 and 25, in the Baker Hotel.

A letter from William H. Lowe, association secretary, gave the theme of the meeting as "the summary of all existing information on the real postwar automotive engines and the motor fuels." Leading technical experts from the automotive, the refining and the natural gasoline fields will appear on one session for an exhaustive presentation.

The "Information Please" session will come along the final day with emphasis on plant problems.

Again the Natural Gasoline Supply Men's Association will have charge of entertainment features, foremost of which will be the buffet-dinner the evening of April 24.

H. W. HARTS

Up For Decision

With the printing of one more chapter, *"The Refinery of the Future,"* the series of articles on basic petroleum chemistry by John J. Stadtherr will be complete. Then an answer can be given as to what shall be done with the scores of requests for reprints.

Recently one inquiry involved a complete training program in a foreign plant. Most of the requests are for a complete set of the 10 chapters, usually from someone who entered a subscription after the series had started.

The whole assortment of inquiries will be studied. Meanwhile others may want to be on a list.

How to reproduce the series and how to distribute the reprint are questions for consideration. Printing facilities continue to be as crowded as hotels. No longer is it practical to instruct the plant to produce a few or many reprints. All too often the time lag is discouraging.

But some disposition must be made of the requests on file in response to the most popular series of articles yet printed in PETROLEUM REFINER.

Studies in Corrosion

Concerning his manuscript, "Corrosion Studies for the Refining Engineer," Professor F. A. Rohrman, Kansas State College, wrote:

"The entire purpose of the paper is to present fundamentals so that the average engineer can understand corrosion. Since my talks before the National Association of Corrosion Engineers in Kansas City and the American Chemical Society in Wichita, I have been deluged with requests for copies of these papers. Just last week I had one from France and one from Russia."

The two papers were responsible for working out a series of three articles, on which type has been set and proof prepared for the author. The three are to be run consecutively, and the month for the first will not be determined until the editorial schedule indicates consecutive use.

Service

For his "exceptionally outstanding service in the development of engine fuels and lubricants for military aircraft," Major John B. Duckworth, assistant director of research of Standard Oil Company (Indiana), was awarded the Legion of Merit by the United States Army Air Forces at the Army air base at Park Ridge, Illinois, March 4.

Major Duckworth's citation reads:

"As test engineer, chief of the fuel unit, and assistant chief, fuel and oil branch, power plant laboratory, headquarters, air materiel command, from August, 1942, to January, 1946, Major Duckworth rendered exceptionally outstanding service in the development of engine fuels and lubricants for military aircraft. His brilliant planning and execution of fuel development projects were contributing factors in the supply of high aviation gasoline to our combat aircraft throughout the world."

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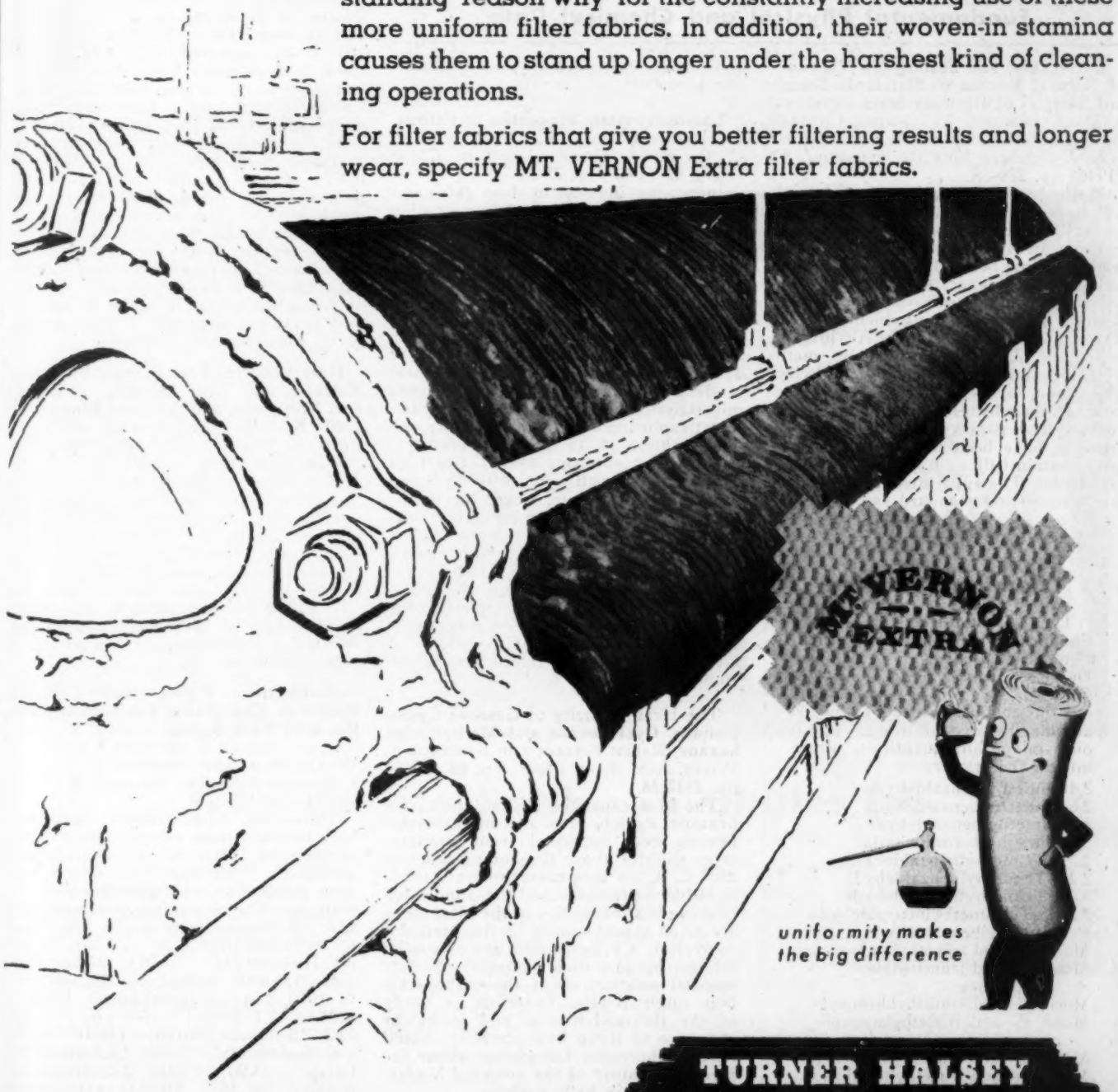

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Traver Road, Ann Arbor, Michigan
under supervision of

DR. E. H. LESLIE and DR. H. B. COATS

The abstracts here presented are selected from the current literature of science and technology to afford reference to fundamental information not easily available to all readers. Abstracts of articles appearing in readily obtainable trade journals are not included.

Photostat copies of original articles will be supplied at cost by The Leslie Laboratories. Complete or limited bibliographies covering special topics by title, by abstracts, or in complete manuscript, will also be prepared and furnished at reasonable cost by the Laboratories.

Fundamental Physical and Chemical Data

Purification and Sealing "In Vacuum" of Natural Bureau of Standards Standard Samples of Hydrocarbons. BEVERIDGE J. MAIR, DOMENIC J. TERMINI, CHARLES B. WILLINGHAM, AND FREDERICK D. ROSSINI, *J. Research Natl. Bur. Standards* **37** (1946) pp. 229-38.

Methods of purification of the standard hydrocarbon samples are described, as well as the procedure for sealing in vacuum for hydrocarbons having normal boiling points below 170° C. and melting below room temperature. Certified samples of n-pentane, isopentane, n-hexane, 2-methylpentane, 3-methylpentane, 2,2-dimethylbutane, 2,3-dimethylbutane, n-heptane, 2,2,4-trimethylpentane, methylcyclopentane, cyclohexane, methylcyclohexane, benzene, toluene, ethylbenzene, 1,2-dimethylbenzene (o-xylene), and meta-zylene, para-xylene, and isopropylbenzene have been prepared. The samples contain 0.01 to 0.36 mol-percent of impurities. The apparatus and procedure for transferring standard samples of C_5 and higher hydrocarbons under vacuum are described.

Chemicals Wanted

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3,5-Dimethylbenzaldehyde
2,3,5-Trimethylbenzaldehyde
2,4,5-Trimethylbenzaldehyde
3,4,5-Trimethylbenzaldehyde
2,3,4,5-Tetramethylbenzaldehyde
Pentamethylbenzaldehyde
Mono, di- and trimethylanthracenes
Mono, di- and trimethylphenanthrenes
Mono, di- and trimethyldiphenyls
Mono, di- and trimethylacenaphthenes
Mono, di- and trimethylfluorenes
Mono, di- and trimethyltetralins
Aryl and alkyl mono- and dicyclohexylamines
Aliphatic and aromatic amine nitrites
Lead tetraoxysulfate
o-n-Butylphenol

Thermodynamic Properties of Ethane. C. H. BARKELEW, J. L. VALENTINE AND C. O. HURD, *Trans. Am. Inst. Chem. Engrs.* **43** (1947) pp. 25-38.

Increasing interest in deep refrigeration for low-temperature fractionation and related operations makes accurate knowledge of the thermodynamic properties of the light hydrocarbons necessary. The paper adds ethane to the list of light hydrocarbons for which reliable thermodynamic properties are available. The literature of the entire subject is reviewed. The authors made use of available data and recalculated the thermodynamic properties. The maximum uncertainties are believed to be as follows: vapor pressure, ±0.5 percent; specific volume, ±0.5 percent; enthalpy, ±1 Btu/lb.; entropy, ±0.001 Btu/(lb.)(°R); activity coefficient, ±0.005: except near the critical state, where the uncertainties are somewhat larger. The results of the calculations are presented in detail in two tables; Thermodynamic Properties of Saturated Ethane, and Thermodynamic Properties of Superheated Ethane. A pressure-enthalpy diagram is included and also a diagram of the compressibility factor for ethane. A bibliography of 17 references is included.

The Heat Capacity of Gaseous Cyclopentane, Cyclohexane and Methylcyclohexane. RALPH SPITZER AND KENNETH S. PITZER, *Jour. Amer. Chem. Soc.* **68** (1946) pp. 2537-38.

The heat capacities of gaseous cyclopentane, cyclohexane and methylcyclohexane were measured from temperatures slightly above the boiling point to 250° C. A few measurements were made at reduced pressures and these indicated that the heat capacities of the ideal gases are given approximately by the Berthelot correction. Cycloparaffins are of special interest because their geometry restricts internal rotation about the carbon-carbon single bonds. Therefore, a study of the thermodynamics and molecular structure of these hydrocarbons should lead to increased knowledge about the form and nature of the potential hindering rotation in hydrocarbons.

Phase Equilibria in Hydrocarbon Systems. H. H. REAMER, B. H. SAGE, AND W. N. LACEY, *Ind. Eng. Chem.* **39** (1947) pp. 77-82.

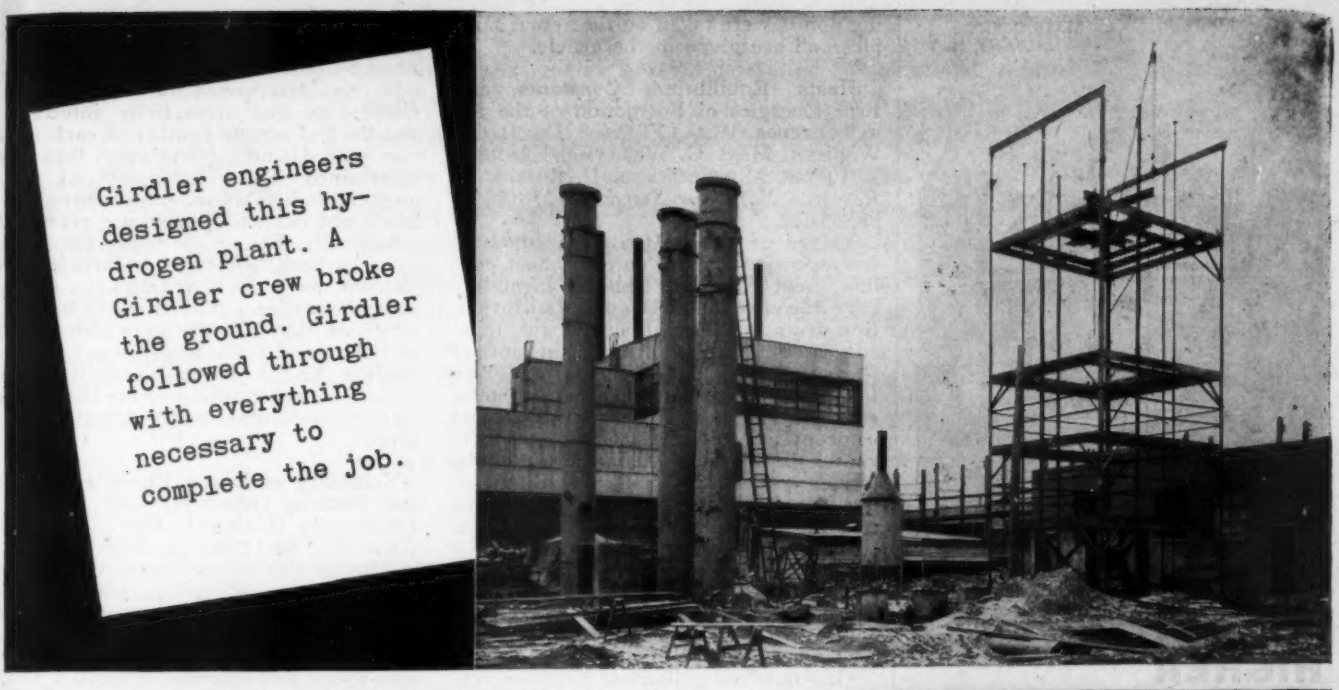
The results of an investigation to establish the volumetric and phase behavior of the system methane-n-butane-decane at pressures up to 10,000 psia and at temperatures between 100° and 460° F. are reported. Five solutions of these hydrocarbons were studied. Concentrations of these solutions were chosen so as to have a fixed molal ratio of n-butane to decane of approximately 2 to 1, whereas the mole fraction of methane was varied from 0.3785 to 0.9775. Results establish the volumetric properties of these solutions in the single-phase region at pressures above that of the bubble point and, in the two-phase region, near the bubble-point boundary. The procedure used in the experimental work is described. The results are presented in detail in tabular and graphical form and a bibliography of 16 references is included.

Heat Content, Free-Energy Function, Entropy, and Heat Capacity of Ethylene, Propylene, and the Four Butenes to 1500° K. JOHN E. KILPATRICK AND KENNETH S. PITZER, *J. Research Natl. Bur. Standards* **37** (1946) pp. 163-71.

The values of the heat-content function, free-energy function, entropy, heat content, and heat capacity are given for ethylene, propylene, 1-butene, cis-2-butene, trans-2-butene, and isobutene in the ideal gaseous state at 298.16° K. and at 100° intervals from 300° to 1500° K. The values were calculated. The calculated values of the heat capacity and entropy were compared with observed values when possible.

Purification, Purity, and Freezing Points of Thirty-One Hydrocarbons of the API-NBS Series. AUGUST R. GLASGOW, JR., ELELYN T. MURPHY, CHARLES B. WILLINGHAM, AND FREDERICK D. ROSSINI, *J. Research Natl. Bur. Standards* **37** (1946) pp. 141-5.

Thirty-one hydrocarbons that had been received from various sources were purified by fractionation or azeotropic distillation. Benzene, and m- and p-xylene were purified by crystallization and centrifuging. Freezing-point determinations and calculations were made. The F.ps. for zero impurity in air at 1 atm. were: n-C_5H_{12}—129.723° ±0.015, 2-methylbutane—159.890° ±0.012, n-C_6H_{14}—95.342° ± 0.010, 2-methylpentane — 153.685° ±0.010, 2,2-dimethylbutane — 99.73° 0.13, 2,3-dimethylbutane—128.41° ±0.06, n-C_7H_{16}—90.595° ±0.010, 2,2,3-trimethylbutane — 24.96° ±0.05, 2,2,4-trimethylpentane—107.365° ±0.013, cyclopentane —93.80° ±0.04, methylcyclopentane—142.445° ±0.012, cyclohexane 6.554° ±0.005, C_6H_6 5.533° ±0.010, toluene—94.991°±0.012, EtPh — 94.950° ±0.025, o-xylene — 25.175° ±0.010, m-lene — 47.872° ±0.020, p-xylene 13.2633° ±0.012, iso-PrPh—96.028° ±0.010. Other freez-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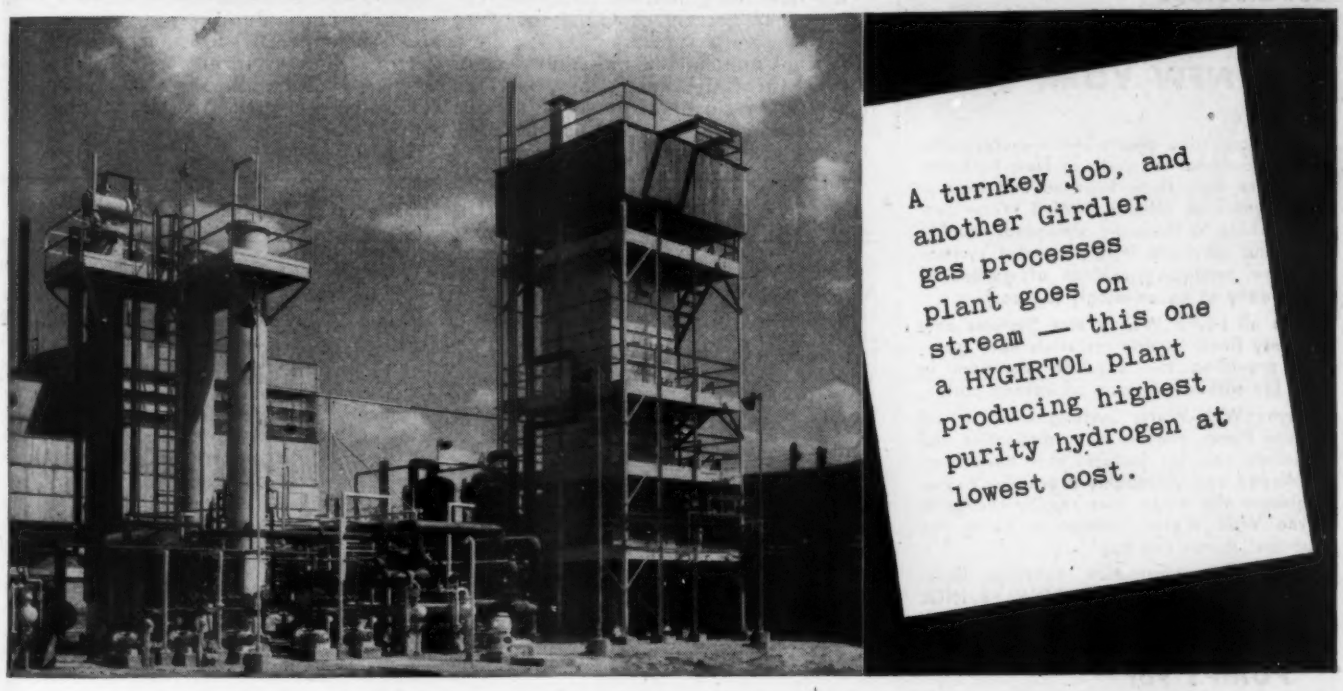
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ing points were taken on the actual samples and are given in the article.

Heats, Equilibrium Constants, and Free Energies of Formation of the Alkylbenzenes. WM. J. TAYLOR, DONALD D. WAGMEN, MARY G. WILLIAMS, KENNETH S. PITZER, AND FREDERICK D. ROSSINI, *J. Research Natl. Bur Standards* **37** (1946) pp. 95-122.

Values of the heat-content function, free-energy function, entropy, heat content, heat capacity, heat of formation from the elements, free energy of formation from the elements, and the logarithm of the equilibrium constant of formation from the elements are given for the hydrocarbons benzene, toluene, ethylbenzene, the 3 xylenes, normal and isopropylbenzene, the 3 methylethylbenzenes, the 3 trimethylbenzenes, and the higher normal alkylbenzenes. Equilibrium constants are presented for some reactions involving alkylation, cyclization, and trimerization. A bibliography of 27 references is included.

Superfractionation Studies. C. C. WARD, R. M. GOODING, AND B. H. ECCLESTON, *Ind. Eng. Chem.* **39** (1947) pp. 105-9.

One hundred gallons of naphtha, representing 12 percent of Oklahoma City crude oil, were superfractionated in a pilot-plant unit having the equivalent of 80 to 90 theoretical plates, and operated at a reflux ratio of about 40 to 1. One hundred and fifty fractions, each representing 0.5 percent of the naphtha, were collected between 73° F. and 258° F. The composition of each of these fractions was determined by use of specific dispersions and refractivity intercepts, and the F-3 octane number of each fraction with 4.0 ml. of tetraethyl lead was determined. The relationships of composition to distillation temperatures, densities, and octane numbers are presented graphically. The octane numbers of pure hydrocarbons with the tetraethyl lead addition were calculated and the results are presented in tabular form. The properties of the iso-cuts were determined and the results are given in a table including ASTM distillation of the fractions. The composition of the iso-cuts in terms of individual hydrocarbons is also given.

Solubility of Boron Fluoride in Normal Pentane. GEORGE N. CADE, ROBERT E. DUNN AND HAROLD J. HEPP, *Jour. Am. Chem. Soc.* **68** (1946) pp. 2454-55.

The solubility of boron fluoride in n-pentane was determined, with an average error of about ±5 percent, at 49, 66 and 93° C. and total absolute pressures ranging from 3.8 to 14.8 atmospheres. It was found to be of the order of 1 to 12 c. c. of boron fluoride per gram of normal pentane. At constant partial pressure of boron fluoride, the solubility was found to be minimum at about 70° C. Results are presented in tabular and graphical form.

Chemical Composition and Reactions

The Preparation and Identification of Alkylcyclopropanes: 1,1,2-Trimethylcyclopropane and 1,2-Dimethyl-3-ethylcyclopropane. JOHN D. BARTLESON, ROBERT E. BURK AND HERMAN P. LANKELMA, *Jour. Amer. Chem. Soc.* **68** (1946) pp. 2513-18.

The Freund reaction for the preparation of cyclopropane by the action of zinc on 1,3-dibromopropane was employed for the preparation of several homologs of cyclopropane. 1,1,2-trimethylcyclopropane was prepared in high yields from 2-methyl-2,4-dibromopentane by the Freund reaction at low temperatures. The principal by-products were found to be 2-methyl-1-pentene and 2-methylpentane. 1,2-Dimethyl-3-ethylcyclopropane was prepared in high yield from 3-methyl-2,4-dibromohexane. An improved method is given for the preparation of secondary and tertiary organic bromides from alcohols or diols.

The Reduction of Unsaturated Hydrocarbons at the Dropping Mercury Electrode. III. Mechanism of the Dimerizing Addition of Sodium to Olefins. STANLEY WAWZONEK AND JOYCE WANG FAN, *Jour. Amer. Chem. Soc.* **68** (1946) pp. 2541-44.

Certain olefins when treated with alkali metals add and dimerize in a manner similar to that shown for 1,1-diphenylethylene. The behavior of unsaturated hydrocarbons at the dropping mercury electrode was found to resemble that observed with alkali metals. Both are sources of electrons with the dropping mercury electrode having the advantage of being a controlled source. The mechanism of the reaction is discussed.

On the Course of the Reaction at 100° C. in the System Paraffin Oxygen. D. J. W. KREULEN, *Jour. Inst. Pet.* **32** (1946) pp. 656-59.

The author investigated the course of the reaction of the oxidation of solid paraffin. The paraffin used had the following physical constants: mol. weight, 418; melting point, 57° C.; aromatic rings, 0 percent; naphthenic rings, 0 percent, paraffins 100 percent. Small quantities of peroxides and aldehydes were present in the paraffin. The quantities of peroxides and aldehydes formed were found to be proportional to the time of oxidation. The quantity of acids was a power function of the time. If copper is present the peroxides are destroyed and an increase in the quantity of aldehydes observed. The results obtained by similarly oxidizing white oils, xylene, and solid paraffin were compared.

Nitroölefins by the Vapor-Phase Catalytic Cleavage of Esters of Nitro Alcohols. MARVIN H. GOLD, *Jour. Amer. Chem. Soc.* **68** (1946) pp. 2544-46.

The literature of the nitroölefins is briefly reviewed. Methods used for the preparation of conjugated nitroölefins by the vapor-phase cleavage of esters of β-nitro alcohols is described. It was found that the acetic acid esters of β-nitro alcohols, and β-chlorinated nitroparaffins undergo cleavage in the vapor-phase with nitroölefins. The action is aided by catalysts such as aluminum phosphate, magnesium phosphate, calcium sulphate and silica gel.

Kinetics of the Diene Sulfur Dioxide Reaction. L. R. DRAKE, S. C. STOWE AND A. M. PARTANSKY, *Jour. Am. Chem. Soc.* **68** (1946) pp. 2521-24.

The reaction of a conjugated diene and

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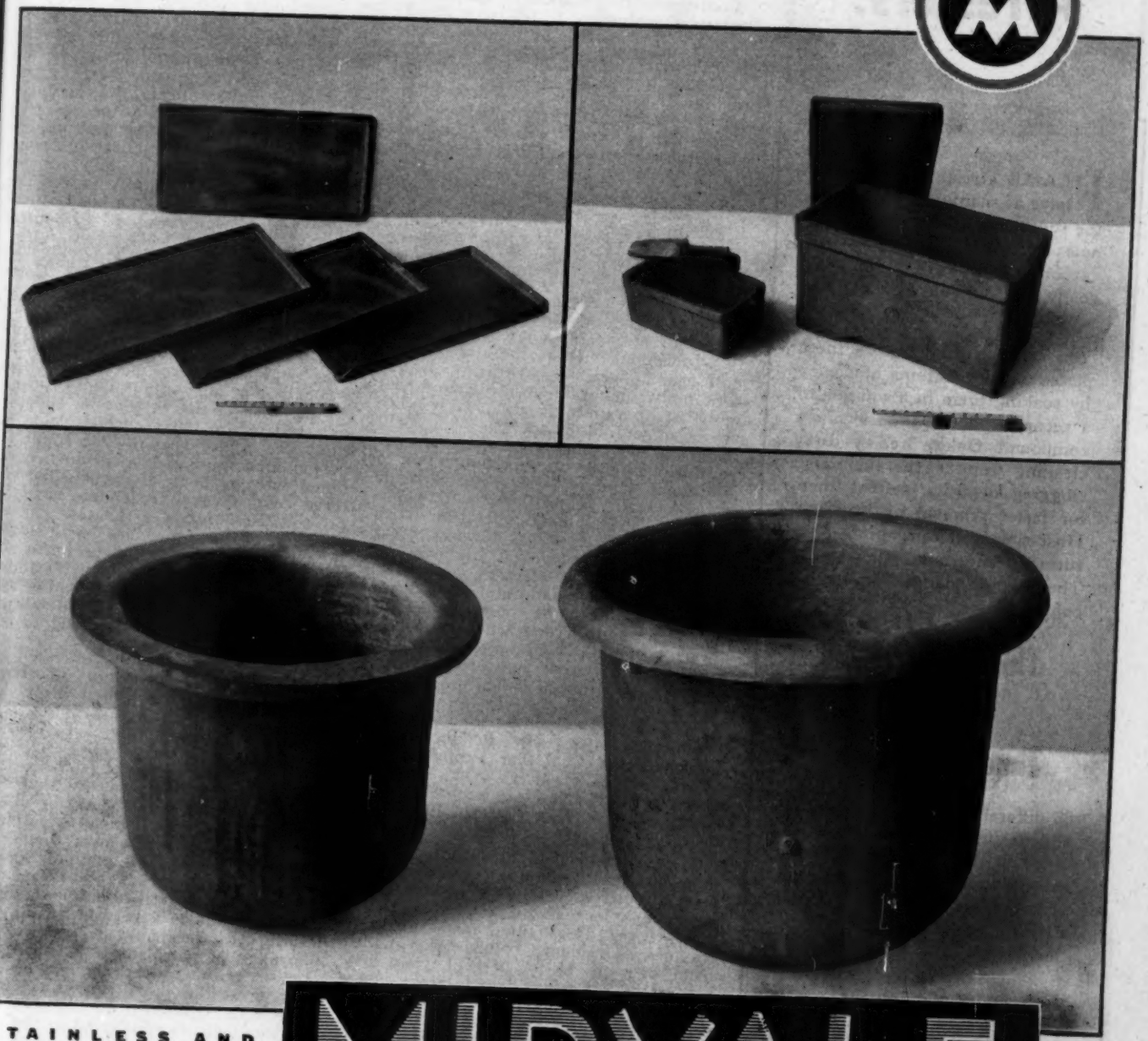

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sulfur dioxide in the presence of an inhibitor to form the monosulfone was found to be an equilibrium reaction. The dienes studied were butadiene, piperylene and isoprene. The heats of formation of the sulfones were measured, as were the energies of activation for the decomposition of the sulfones. Data are given in detail in tabular form and are presented graphically.

Heat Transfer Coefficients for Condensing Hydrocarbon Vapors. DANIEL A. DONOHUE, *Ind. Eng. Chem.* **39** (1947) pp. 62-4.

The theoretical Nusselt equation can be reduced to the form $h_m = C(L/w)^{1/3}$ in which C is a constant, L is the horizontal tube length, in feet, and w is the rate of flow of condensate from the lowest point on the condensing surface, as lb./hr. All of the other terms in the Nusselt equation are combined into the constant C. Over the usual condensing temperature range for hydrocarbons the values of the constant C vary between 700 and 800 for natural gasoline, between 667 and 704 for gasoline and between 621 and 684 for kerosine. Since the solution requires that only the condensate loading w/L be known, the necessity for determining values of the physical properties of the hydrocarbons at the pertinent film temperature is unnecessary. The application of the equation to the commercial design of condensers is illustrated.

Manufacture: Processes and Plant

Continuous Preparation of Butadiene-Styrene Copolymer. J. J. OWEN, C. T. STEELE, P. T. PARKER, AND E. W. CARRIER, *Ind. Eng. Chem.* **39** (1947) pp. 110-13.

The continuous preparation of Buna-S was studied in one-stage, three-stage, and five-stage systems in the laboratory. Multi-stage procedure was used to minimize "short-circuiting." Runs of 45 to 618 hours were made. The number of stages, percentage conversion, amount and manner of catalyst and modifier addition, hydrogen ion concentration, and ratio of water to hydrocarbon were the principal variables studied. The data indicate that under comparable conditions and at the same extent of conversion the products from the one-, three-, and five-stage operation showed similar properties. Increasing the number of stages did not show appreciable advantage. Under conditions comparable to those employed in batch operation, reaction rates were slower in continuous operation but could be increased by raising the reaction temperature or by the interstage injection of additional catalyst. The Buna-S made was of quality equivalent to that obtained in typical batch operation when the conversion level was lowered or at higher conversion levels if modifications were made in the reaction conditions, including interstage addition of modifier, interstage addition of catalyst, decreasing the water to hydrocarbon ratio, and lowering of the pH of the soap solution. Results of this study indicate that continuous production of Buna-S is entirely feasible.

Experiments on the Production of Toluene from Coal Tar Products and Other Sources. C. M. CAWLEY, J. H. G. CARLILE, H. E. NEWALL, AND F. E. T. KINGMAN. *Jour. Inst. Pet.* **32** (1946) pp. 660-83.

The article reports results of work done in Great Britain during the war to meet the increased toluene requirements. Benzene, xylene, cresol, and naphthalene were viewed as potential raw materials for making toluene. Toluene was made from benzene in 94 percent yield by preparation of benzaldehyde from benzene and carbon monoxide in the presence of aluminum chloride, followed by the hydrogenation of benzaldehyde to toluene. It was found that the xylene in solvent naphtha could be converted into toluene by cracking over silver gauze to give a yield of 38 percent in one stage. If pressure above atmospheric was used in an atmosphere of hydrogen the conversion reached 40 to 50 percent. Particular attention was paid to the preparation of toluene by the hydrogenation of cresol. A continuous process was developed working at a pressure of 10 to 20 atmospheres and a temperature of 430-440° C. A semi-commercial plant was built. The yield of toluene was 87.3 percent of the theoretical. Methyl-cyclohexane to the extent of 7.3 percent also was made. Normal heptane was used as a raw material for cyclization, using chromic oxide supported on activated alumina as the catalyst. At a reaction temperature of 550° C., the yield of aromatic hydrocarbons was 57 percent of the heptane treated.

Estimating Chemical Piping Costs. R. J. SCHRADER. *Chem. Eng.* **54** (1947) pp. 109-10.

Pipe material pricing data are presented in tabular form for standard pipe, stainless-steel pipe, pyrex pipe, porcelain pipe, and saran piping. Fittings are included in this tabulation. In order to estimate the labor costs of pipe fittings, the author presents a formula for determining piping labor which combined with per-hour labor costs and material costs gives a reasonable basis for pre-construction cost estimates. The application of the procedure is illustrated by a typical example. If working conditions are cramped, 15 percent is added. If new piping ties into an existing system, 15 percent is added. If no fabrication is permitted on the job, add 10 percent.

Catalytic Desulfurization of High-sulfur Stocks by the Cobalt Molybdate Process. C. BERG, W. E. BRADLEY, R. I. STIRTON, R. G. FAIRFIELD, C. B. LEFFERT, AND J. H. BALLARD. *Trans. Am. Inst. Chem. Engrs.* **1** (1947) pp. 1-12.

Methods of sulfur removal that have been used in the industry are briefly reviewed, including chemical treatments and contact treatments over oxide-type catalysts. None of these methods remove the refractory thiophenes and thiophanes. The process described utilizes cobalt molybdate and is a versatile method of desulfurization capable of handling the full range of petroleum stocks encountered in refining operations and to remove even the most refractory sulfur compounds contained in these stocks. The treatment is conducted in the presence of hydrogen. Pilot-plant studies have shown the process to be effective in handling stocks containing 5.0 percent or more of sulfur. Sulfur contents of the products vary from 0.1 percent for light distillates to 0.5 percent for the heaviest distillates, including material in the lubricating oil boiling range. The

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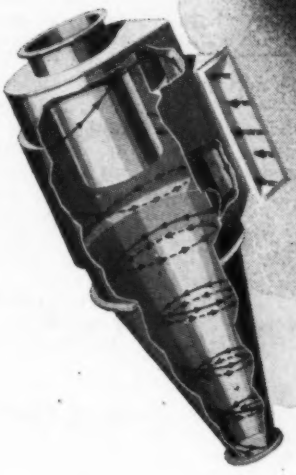

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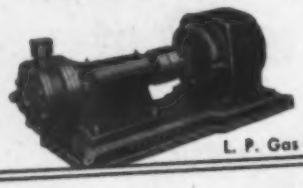
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important variables have been evaluated, such as the effect of hydrogen sulfide in the hydrogen gas, and the effect of temperature, pressure, space velocity, and hydrogen rate. Most of the work has been done with light and heavy distillates produced by coking Santa Maria Valley crude. The pilot plant used in the work is described and the data are presented in considerable detail in tabular and graphical form.

Acetone Direct from Acetylene. A. W. J. Dyck. *Can. Chem. Process Inds.* **30** (1946) pp. 34-6.

A new process for the synthesis of acetone is described. The original process used by Shawinigan Chemicals, Ltd., involved six stages. The present process involves the direct reaction of acetylene and steam in the presence of a catalyst, which contains a mixture of ferric oxide and zinc oxide. The Fe_2O_3 is prepared by treating a 45 percent solution of ferric chloride with ammonia. The reactor comprises a series of vertical tubes properly spaced and grouped in a cylindrical unit. The reaction mixture enters the tube from the bottom. A diagrammatic flow sheet of the plant is given.

Products: Properties, Utilization and Analysis

Tendency to Smoke of Organic Substances on Burning. Part II. Smoke Production and Burning Characteristics of Hydrocarbon Gels. F. H. Garner, T. G. Hunter, and A. E. Clarke. *Jour. Inst. Pet.* **32** (1946) pp. 643-55.

During the war hydrocarbon gels were widely used as filling materials for incendiary bombs. Also thickened kerosine has been used for beacons, flares and as a source of emergency heat. Smoke production from various hydrocarbon gels was measured. The methods employed and the results obtained are described in the paper. The effect of the following variables was studied: Chemical composition of the hydrocarbon base; volatility of the hydrocarbon base, and viscosity of the gel. To reduce smoke formation, it was found the composition of the hydrocarbon base was most important. Volatility was of equal importance in controlling the burning characteristics. The viscosity of the gel had no apparent effect on smoke production. The more saturated or paraffinic the hydrocarbon base the less smoke generation. The use of a gel prepared from an aromatic-free saturated-hydrocarbon base results in a marked reduction in smoke formation. The aromatics, such as benzol, are particularly bad as smoke producers.

Quantitative Determination of Paraffins and Naphthenes in Gasolines. S. G. Hindin and A. V. Grosse. *Anal. Chem.* **19** (1947) pp. 42-47.

The authors recently described a lamp method for the accurate determination of the hydrogen content of liquid organic compounds and mixtures, particularly in saturated hydrocarbons. The present paper discusses the application of this technique to the estimation of paraffins and naphthenes in gasolines, after removal of aromatics and olefins by any of the usual procedures. In view of the increased use of the various catalytic processes, the quantitative estimation of paraffins and naphthenes has been assuming greater importance. The accuracy of the procedure described is independent of isomeric composition; it varies from ± 1.5 weight-percent paraffin in the C_5 cut up to ± 3.0 percent in the C_{12} cut. The application and use of the hydrogen-to-carbon ratio in the solution of various analytical problems is suggested. The data secured in the course of the work are presented in considerable detail in tabular and graphical form.

Determination of Aromatics and Naphthenes in Complex Hydrocarbon Mixtures Containing Olefins. R. M. Love, A. R. Padgett, W. D. Seyfried, and H. M. Singleton. *Anal. Chem.* **19** (1947) pp. 37-42.

A simple and rapid method of analysis for the naphthenes and aromatics boiling in the range of 93° to 149° C. in naphthas containing up to 40 percent of olefinic compounds was required in the course of the work at the Baytown Ordnance Works in which toluene was produced from petroleum. None of the methods described in the literature were regarded as suitable and this led to the development of the method described. Interfering olefins and diolefins, if present, were removed by bromination and steam-distillation; after fractionation of the steam-distillate, the aromatics and naphthenes were determined in selected fractions by taking refractive indices before and after refraction with sulfuric acid. The method was found to be accurate to about ± 0.3 percent on aromatics and ± 1.0 percent on naphthenes. The method has certain limitations and will not reveal such compounds as unsaturated naphthenes, since they are removed in the bromination and steam-distillation step. It will not distinguish between the C_8 aromatics, nor between naphthenes of the same boiling range, such as methylcyclohexane and ethylcyclopentane, nor between the various dimethylcyclohexanes. However, the presence of conjugated diolefins, which are troublesome in other methods of analysis, does not affect the results obtained by this method.

Determination of Traces of Fluorine in Organic Compounds. W. B. Huckabay, R. H. Busey, and A. V. Metler. *Anal. Chem.* **19** (1947) pp. 59-63.

A method is given for determining very small amounts of fluorine as organic fluoride in the solutions of other organic substances. The sample is vaporized and burned from a jet. Combustion is completed by passing the products over hot platinum in the presence of oxygen. After collection in an absorption tower, the fluorine is determined volumetrically by a combination of conventional methods. Results obtained are superior to those obtained by other current methods, in that the decomposition of the fluoride is more complete than when the material is simply burned from a jet. Sensitivity of the method is limited only by the size of the sample. Data are given for work on samples of known fluorine content. The general method of decomposition is applicable to analogous cases when the sample contains traces of organic compounds of the other halogens and possibly sulfur. Data are given for organic bromides. The apparatus used in the work is described in considerable detail and the procedure is given. A bibliography of 18 references is included.

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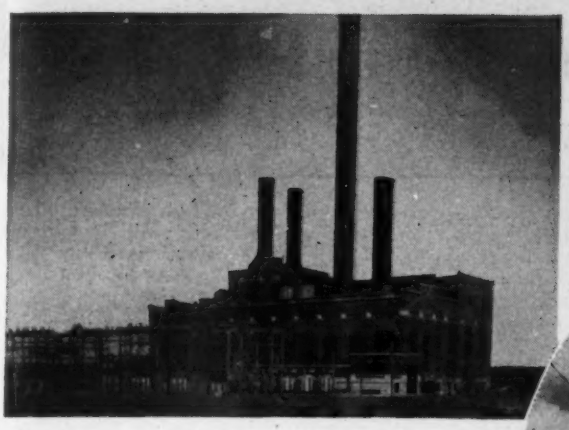

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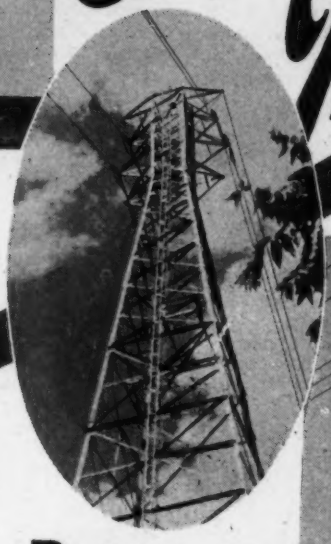

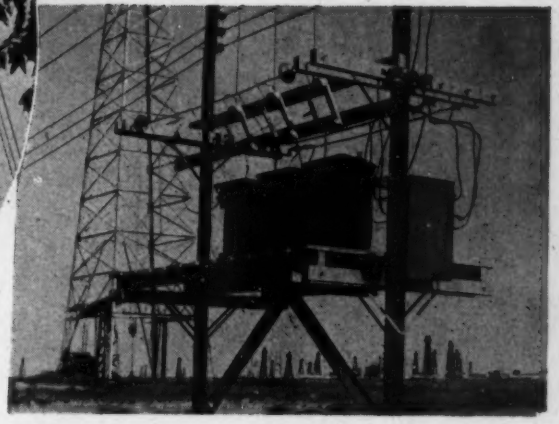

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REFINING

U.S.P. 2,412,765. Recovery of Hydrocarbons. E. Buddrus and S. C. Carney to Phillips Petroleum Company.

Petroleum oil is produced from partially depleted wells or oil-bearing formations by injecting condensible hydrocarbon vapors consisting of propane and butane at atmospheric temperature and at a maximum pressure only slightly higher than the vapor pressure of propane at formation temperature into the partially depleted oil-producing or bearing formation. The injected vapors are permitted to condense within the pores of the formation, thereby warming by the available heat of condensation the residual crude oil and dissolving this residual oil. The mixture of fully condensed propane, butane and petroleum oil is fractionated into a crude oil bottoms and propane and butane vapor. This vapor is recycled into the partially depleted oil-producing or bearing formation.

U.S.P. 2,412,791. Method for Purifying Mineral Oils. L. C. Waterman to Petrolite Corporation, Ltd.

Water-dispersable impurities are removed from a stream of crude oil flowing at a non-uniform rate by mixing with this stream a stream of relatively fresh water to form a stream of oil-continuous emulsion in which droplets of water coexist with water-dispersable impurities. This stream of emulsion is divided into a plurality of separate streams which are delivered to a plurality of separate high-intensity electric fields in separate settling spaces disposed at the same level. The dispersed water is coalesced, and the water-dispersable impurities are brought into association therewith. The electrically treated constituents of each separate stream are separated in its corresponding settling zone to produce therein bodies of purified oil and water which contains most of the impurities. Streams of water are simultaneously withdrawn from each of these zones and are combined. The rate of flow of water in the combined stream is controlled in accordance with changes in the total amount of separated water in only one of the separating zones and in a manner to maintain the total amount of separated water in this zone substantially constant. Streams of purified oil are withdrawn from the upper ends of the separate settling zones and are combined. This combined oil stream is throttled in response to changes in pressure of this stream and in a manner to maintain the pressure in the settling zones substantially constant.

U.S.P. 2,412,823. Furfural Recovery. B. J. Mayland to Phillips Petroleum Company.

Furfural used as an extracting agent for unsaturated hydrocarbons is recovered from its admixture with hydrocarbons having boiling points so close to that of furfural as to make separation by distillation difficult, by subjecting the mixture to water extraction in the presence of a liquid hydrocarbon having a boiling point substantially lower than that of furfural. Preferably a hydrocarbon of not more than 8 C atoms, such as n-pentane, is employed.

U.S.P. 2,412,880. Azeotropic Distillation of Butadiene and 2-Butene. F. E. Frey to Phillips Petroleum Company.

Butadiene is separated in a highly concentrated form from other C_4 hydrocarbons by subjecting a hydrocarbon material essentially comprising n-butane, 1-butene, 2-butene, and butadiene, with the proportion of 2-butene being greater than the proportion of butadiene, to fractional distillation in the presence of SO_2 in an amount sufficient to form minimum-boiling azeotropes with all of the n-butane and 1-butene present but insufficient to form an azeotrope with the major portion of the 2-butene present. A separation of 2-butene and butadiene as a kettle product substantially free of other C_4 hydrocarbons is thus effected. The kettle product is subjected to a second fractionation in the absence of SO_2 to separate concentrated butadiene.

U.S.P. 2,413,185. Removal of Volatile Metal Halides from Fluids. W. A. LaLande, Jr., to Porocel Corporation.

Volatile metal halide, such as $AlCl_3$, is removed from a non-aqueous fluid containing the same particularly from a hydrocarbon fluid by contacting the fluid with an alkali metal acid sulfate which is converted to alkali metal sulfate, metal sulfate and hydrogen halide. The treated fluid containing hydrogen halide is separated from the sulfates, and the hydrogen halide is then removed from the fluid.

U.S.P. 2,413,245. Azeotropic Distillation of Toluene. H. C. Reed and B. M. Holt to Union Oil Company of California.

Aromatic hydrocarbons, such as toluene, are separated from non-aromatic hydrocarbons of similar volatility by distilling the mixture of hydrocarbons in the presence of an azeotrope-former having a volatility similar to that of the hydrocarbons, such as methyl ethyl ketone, and a solvent of lesser volatility which is substantially insoluble in the hydrocarbons and has a higher molal heat of vaporization than the azeotrope-former, e.g., water. The distillate comprises substantially all of the non-aromatic hydrocarbons, the major proportion of the azeotrope-former and solvent. The bottoms fraction comprises substantially pure aromatics and a minor proportion of azeotrope-former and solvent. The distillate is cooled and condensed. It separates into two phases, a solvent phase containing a major proportion of solvent and a minor proportion of azeotrope-former, and a hydrocarbon phase consisting predominantly of non-aromatic hydrocarbon and azeotrope-former. The latter is separated from the hydrocarbons by a process involving selective solution of the azeotrope-former in said solvent. The azeotrope-former is separated from the solvent by a distillation process in which all of the azeotrope-former is vaporized and distilled together with a portion of the solvent. The ratio of solvent to azeotrope-former in the distillate is greater than that in the distillate of the first named distillation, while pure solvent is left as bottoms. The distillate comprising azeotrope-former and solvent is recycled to the azeotropic distillation step.

U.S.P. 2,413,253. Purification of Indene. F. J. Soday to The United Gas Improvement Company.

Indene derived from light oil is purified by commingling the impure indene with less than 5 percent by weight of finely divided metals or alloys of metals of groups I A or II A of the periodic system, such as sodium, potassium, or calcium. The indene is recovered from the resulting mass in a purified form.

U.S.P. 2,413,254. Process for Refining Unsaturated Hydrocarbon Material with an Alkali or Alkaline Earth Metal. F. J. Soday to The United Gas Improvement Company.

A mixture of olefinic material containing acetylenic material is continuously passed in a vapor phase at a temperature below 100° C. through a dispersion of a finely divided metal or alloy of metals of groups IA or IIA of the periodic system. The dispersion contains less than 30 percent by weight of the finely divided metal or alloy and is at least one foot in thickness in the direction of flow of the mixture. The concentration of the olefin material in the reaction zone is maintained below 80 percent by weight of the total material present. The rate of flow per hour of the mixture through the dispersion is maintained at less than four times the weight of dispersion medium employed. Olefin material less contaminated with impurities, such as acetlyenic material is continuously removed from the reaction zone sufficiently rapidly to prevent a large loss of olefin by polymerization.

U.S.P. 2,413,255. Process for Refining Styrene and Methyl Styrene. F. J. Soday to The United Gas Improvement Company.

A hydrocarbon mixture containing styrene or methyl styrene and impurities including acetylenic compounds is treated in the manner described in U.S.P. 2,413,254 to obtain refined styrene or methyl styrene.

U.S.P. 2,413,256. Chemical Process and Product. F. J. Soday to The United Gas Improvement Company.

The process of U.S.P. 2,413,254 is used for refining a light oil diolefine fraction

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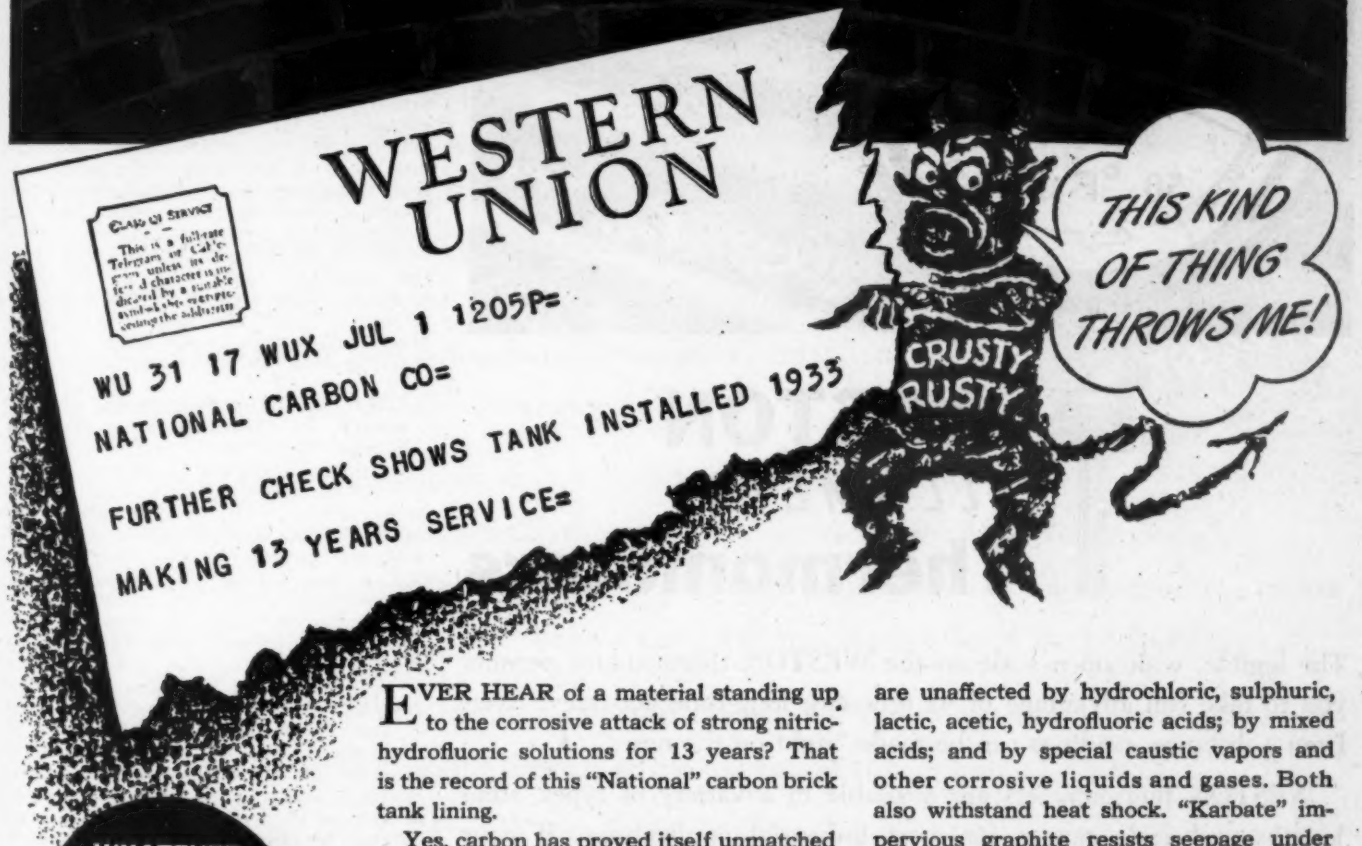
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containing acetylenic impurities, particularly a butadiene fraction.

U.S.P. 2,413,257. Process for Purifying Piperylene. F. J. Soday to The United Gas Improvement Company.

The process of U.S.P. 2,413,254 is used for the refining of piperylene containing corresponding impurities.

U.S.P. 2,413,260. Refining of Aromatic Hydrocarbons with Acid-Acting Metallic Halides. F. J. Soday to The United Gas Improvement Company.

Aromatic hydrocarbon material boiling below 400° C. and contaminated with polymerizable impurities of similar boiling points is contacted with an acid-acting metallic halide catalyst under conditions causing the formation of polymer from a portion of the impurities. The catalyst is then neutralized and the aromatic hydrocarbon material is separated from the resulting reaction mass including the polymer. The aromatic hydrocarbon material is then again contacted with additional acid-acting metallic halide catalyst to form additional polymer, and the reaction mass is treated as before to obtain refined aromatic hydrocarbons.

U.S.P. 2,413,262. High-Compression Motor Fuel. R. I. Stirton to Union Oil Company of California.

The patent relates to an aviation-type gasoline having a compatibility temperature below about —50° F. and which comprises a blend of 0.5-15 percent of an amine with 7-12 C atoms, such as xylidine or N-methyl aniline. The blend further comprises more than 5 percent of aromatic hydrocarbons with 7-10 C atoms, and a mixture of hydrocarbons boiling in the gasoline range other than said aromatic hydrocarbons with 7-10 C atoms. More than 2 ml. per gallon of tetraethyl lead are added. The final blend has an anti-knock rating at least equal to that of 2,2,4-trimethyl pentane.

U.S.P. 2,413,310. Process for the Recovery of Hydrocarbons from a Sludge. H. S. Bloch to Universal Oil Products Company.

A sludge formed by the treatment of hydrocarbons with a metal halide or a hydrogen fluoride catalyst is commingled with an aqueous hydrolyzing medium and a low-boiling substantially inert hydrocarbon liquid. The sludge is hydrolyzed while the hydrocarbon liquid is refluxed to dissipate the exothermic heat of hydrolysis as the heat of vaporization of this liquid and to maintain the hydrolysis temperature at the boiling point of the liquid. Hydrocarbons liberated from the sludge by hydrolysis are recovered from the reaction mixture. Water or dilute acid, alkali or salts can be used as the hydrolyzing medium, and butane, pentane, or a naphtha fraction boiling as high as about 200° C. can be employed as the hydrocarbon liquid to be refluxed.

U.S.P. 2,413,482. Diesel Type Fuel. B. T. Anderson and M. T. Flaxman to Union Oil Company of California.

The patent relates to a highly refined fuel of the diesel type deficient in lubricating properties which contains a small proportion of a lubricity agent soluble in the fuel. The proportion of this agent is sufficient to impart lubricating characteristics to the fuel but is present in an amount of less than about 1 percent. The lubricity agent comprises an extract from a petroleum fraction boiling above

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300° F. and containing phenol homologs and naphthenic acids. An aqueous sodium hydroxyde solution is employed for extracting the petroleum fraction, and the aqueous extract thus obtained is acidified to release said phenol homologs and naphthenic acids.

U.S.P. 2,413,503. Absorption Process. D. La Verne Katz to Phillips Petroleum Company.

Desirable constituents are separated from the gaseous effluent of a condensate well within the pressure range of 3000-6000 psi by contacting the gas at a first point with a first absorber oil of average mol weight 180-280 and further contacting the gas at a second point with a second absorber oil of low viscosity and average mol weight 300-600 to remove vapors of the first absorber oil. The two absorber oils are mixed at said first point. The absorbed desired constituents are stripped from the mixed oil. A portion of the stripped mixed absorber oil is returned to the first contacting step. The remaining portion of this oil is separated into a heavy reisdue and low-boiling constituents. The latter are fractionated to separate lower-boiling constituents from the low-viscosity high-boiling absorbent which is reused as the second absorber oil.

U.S.P. 2,413,674. Multistage Method of Purifying Styrene. H. M. Weir to The United Gas Improvement Company.

A mixture of styrene with xylenes including o-xylene is fractionated to produce a fraction richer in styrene than any eutectic composition capable of being formed between styrene and other constituents of this fraction. The fraction is then subjected to fractional crystallization by cooling to produce a mass comprising styrene crystals and liquid-phase material richer in xylene than said fraction and richer in styrene than any said eutectic composition. The liquid-phase material is separated from said mass to leave a remaining mass containing xylene but richer in styrene than said fraction. This remaining mass is fractionally distilled to further concentrate styrene therein. From a light oil material produced in the manufacture of combustible gas by a process involving the pyrolysis of petroleum oil a fraction containing 45-80 percent styrene can be obtained by the first fractional distillation. The final product of this process then contains more than 95 percent styrene.

U.S.P. 2,413,868. Removal of Organic Fluorine. F. E. Frey to Phillips Petroleum Company.

Hydrocarbon material containing a minor quantity of organically combined F, as obtained for example in the conversion of hydrocarbons in the presence of a HF catalyst, is subjected to the action of a granular product resulting from treating bauxite with a solution of HF in a liquid paraffinic-hydrocarbon stream at a temperature of 50-350° F. for a time such that the bauxite takes up substantially more HF from the hydrocarbon solution than stoichiometrically corresponds to the Al in the bauxite. This action is effected at a temperature and for a time such that no extensive chemical changes in the hydrocarbon material itself are effected but that the organic fluorine compounds are decomposed to fluorine-free compounds and free HF.

U.S.P. 2,413,871. Process for Removing Organically Combined Chlorine from Hydrocarbons. H. J. Hepp to Phillips Petroleum Company.

Hydrocarbons containing organically combined Cl are treated with a mixture of alumina, e.g., dehydrated bauxite, and quicklime under conditions such as to effect decomposition of at least a major proportion of the organic Cl compounds to form HCl and combination of this HCl with the quicklime. The conditions are also such that no extensive chemical changes in the hydrocarbons are effected. The mixture can, for example, contain 5-25 percent of quicklime. A temperature of 300-900° F. and a pressure ranging from atmospheric to 100 psi can be used for effecting the decomposition of at least 85 percent of the organic chlorine compounds present.

U.S.P. 2,413,938. Refining Hydrocarbon Distillates. C. E. Adams and T. B. Tom to Standard Oil Company (Indiana).

Hydrocarbon distillates containing weakly acidic substances are contacted with an aqueous solution of an alkali metal hydroxide and a solutizer comprising an alkali metal salt of a carboxy ether with 3-8 C atoms. The process permits, e.g., the removal of mercaptans from hydrocarbons. The aqueous solution employed contains, for example, KOH and an alkali salt of propoxy acetic acid.

U.S.P. 2,413,945. Treating Petroleum Distillates. J. A. Bolt to Standard Oil Company (Indiana).

A gasoline substantially sweet to the doctor test is obtained from sour gasoline by extracting a major proportion of the mercaptans from the sour gasoline. Remaining mercaptans are oxidized to disulfides in the presence of a phenolic mercaptan oxidation catalyst. A strong caustic alkali solution containing a solutizer is conducted through the oxidation zone and then through the extraction zone, countercurrent to the flow of gasoline. The caustic solution separated from the gasoline in the extraction zone is regenerated by oxidizing a major proportion of dissolved mercaptans to disulfides insoluble in this solution. The disulfides are separated and the solution is then returned to the oxidation zone, an oxygen-containing gas being supplied to this zone to complete the conversion of residual mercaptans.

CATALYST PREPARATION ACTIVATION AND REGENERATION

U.S.P. 2,412,696. Process for Regeneration of Cracking Catalysts. J. R. Schonberg and D. L. Campbell to Standard Oil Development Company.

A hot freshly regenerated catalyst, such as an acid-treated clay, is suspended in a free-oxygen-containing gas and cooled while in suspension. This cooled suspension is mixed with a suspension of spent catalyst in a corresponding gas. The mixture is discharged into a regeneration zone and maintained therein in a highly agitated state by causing it to flow in a multiplicity of vari-directional currents and for a sufficient time to effect the desired regeneration. A portion of the total hot regenerated catalyst recovered is resuspended in additional

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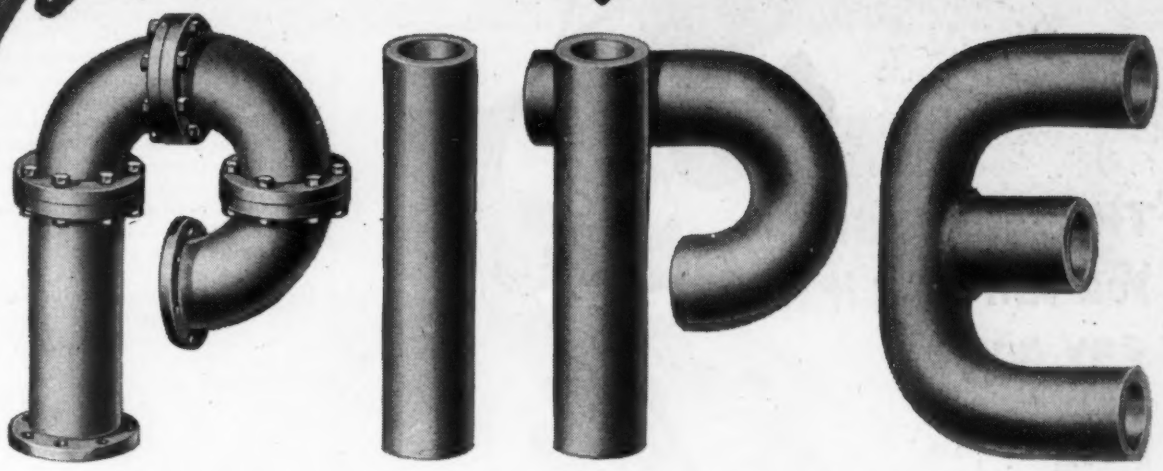

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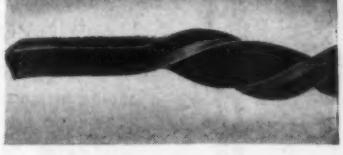

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free-oxygen-containing gas and added to the spent catalyst in the aforesaid manner.

U.S.P. 2,412,879. Chemical Process. H. G. M. Fischer to Standard Oil Development Company.

Frangible and easily disintegrating coke is continuously produced by coil preheating heavy petroleum oil to coking temperature without substantial coke formation. The preheated oil is mixed with a cellulosic material, such as wood sawdust, and this mixture is passed into a zone wherein the coke is formed from the oil in intimate admixture with the cellulosic material or its carbonization products, such as charcoal. The coke formed is continuously disintegrated adjacent a point of its withdrawal from the coking zone.

U.S.P. 2,412,958. Production of Solid Contact Material. J. R. Bates and H. A. Shabaker to Houdry Process Corporation.

A freshly formed inorganic gel is continuously supplied in wet condition to an ageing zone and moved through this zone at a controlled rate to form a moving layer of substantial depth sufficient to retain imparted temperature. The gel is heated from within the layer to rapidly raise the temperature thereof to 120-200° F. The moving layer is retained in an atmosphere of steam and air at a temperature in this range for a period not in excess of 2 hours to effect ageing of the gel without substantially drying it. The aged gel is useful as a contact material.

U.S.P. 2,413,184. Metal Aluminate Adsorbents and Method of Making Same. W. A. LaLande, Jr., to Porocel Corporation.

An alkali metal aluminate and a water-soluble salt of a metal capable of forming a water-insoluble metal aluminate, such as $MgCl_2$, are reacted in aqueous solution in the presence of a compound yielding NH_4+ ions in this solution, such as ammonium chloride. The insoluble aluminate formed is separated, washed and dried. It is employed as a decolorizing adsorbent and as a catalyst.

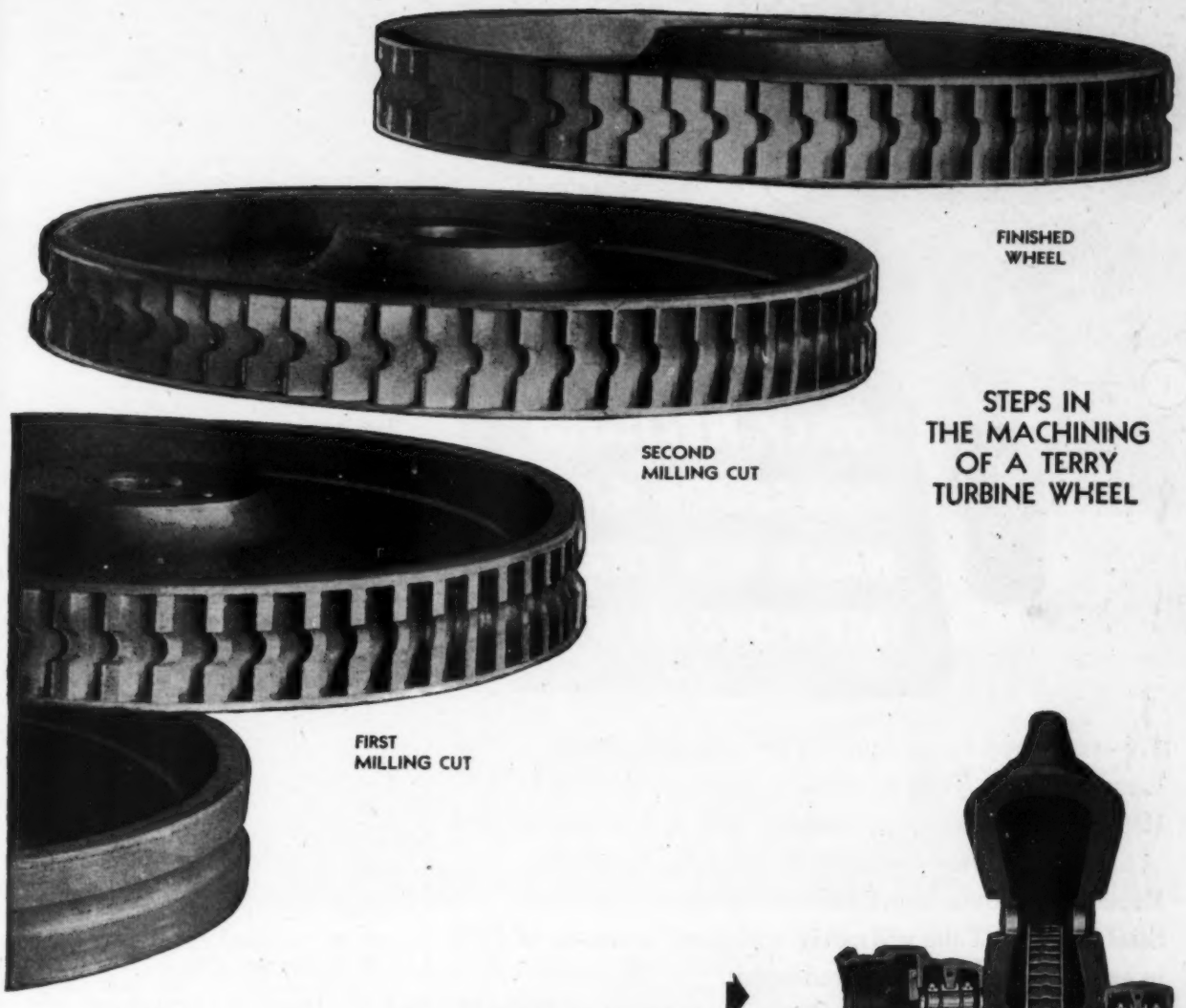
U.S.P. 2,413,961. Pelleting of Catalysts. W. G. Evans to Universal Oil Products Company.

Powdered adsorbent and catalytic materials, such as materials comprising silica gel, silica and alumina, or kieselguhr and a reducible nickel compound, are pelleted in admixture with a relatively small amount of a lubricant consisting essentially of wood rosin. The resultant pellets are heated to remove the rosin. Wear and breakage of the dies and punches of the pelleting machine are reduced by the addition of the lubricant, and pellets of relatively low desnity and high thermal stability are obtained.

U.S.P. 2,414,002. Regeneration of Subdivided Solid Contact Material. C. L. Thomas and J. T. Pinkston to Universal Oil Products Company.

Subdivided solid catalyst particles, such as used in hydrocarbon conversion processes, are regenerated and freed from combustible contaminants by maintaining a first and a second relatively dense fluid-like bed of the solid particles at combustion temperature. Contaminated catalyst particles are commingled with a stream of oxidizing gas and supplied upwardly into the first bed by the gas-lifting action of the oxidizing

Wheel Turbine



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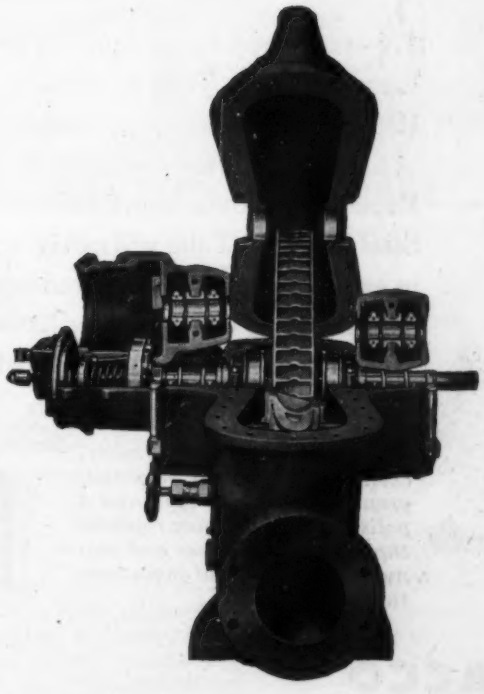

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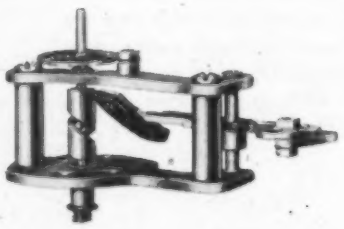

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gas. The gas is passed through this bed at a sufficiently low rate that substantially its entire free oxygen content is consumed in burning combustibles within the bed. Partially regenerated particles are removed from the first bed at a point below the upper extremity of the bed and are supplied by gravity and in the form of a relatively dense column to the upper portion of the second bed. A second independent stream of oxidizing gas is passed upwardly through this bed at a sufficiently high rate that only a portion of its free oxygen content is consumed within the bed. Passage of the gaseous products from the second bed through the first bed is prevented. A stream of regenerated catalyst particles from the second bed is removed at a relatively low point thereof, so that the general direction of flow of the solid particles in this second bed is countercurrent to the oxidizing gas passed therethrough.

CRACKING AND REFORMING

U.S.P. 2,412,837. Manufacture of Liquid Hydrocarbons Suitable for Motor Oil. J. R. Rose to Potomac Hydrocarbon Process Corporation.

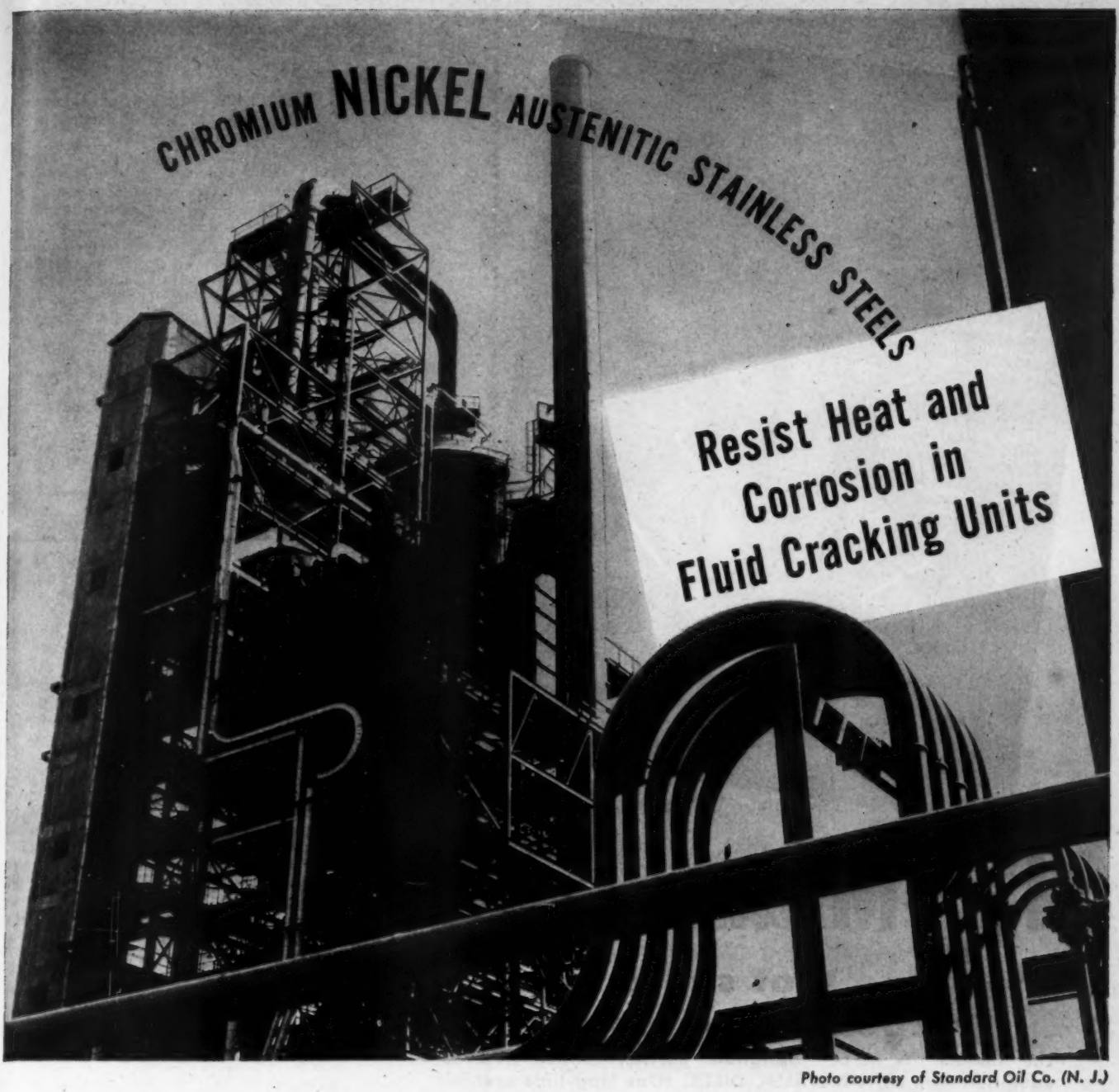
A hydrocarbon-base fluid is subjected to a pressure of 700-1000 psi. The fluid is cooled and conducted under this pressure in an upward direction through a vertically arranged series of cracking elements and a catalyst container. Both the cracking elements and the catalyst container are heated by a heating medium contacting first the lowermost cracking element and thereafter the higher cracking elements and the catalyst container and producing an initial temperature of 700-1200° F. in the lowermost of the cracking elements. The gaseous product from the catalyst container is delivered to a second catalyst container located above the first one and heated by the medium which has first contacted the cracking elements and the first catalyst container. The gaseous product is liquefied by sudden reduction of pressure and by cooling. The liquid obtained is stabilized to evolve hydrocarbon gases and heavy liquid hydrocarbon polymers. The product is suitable for use as a motor fuel.

U.S.P. 2,412,868. Catalyst Cracking of Hydrocarbon Oils. C. L. Brown to Standard Oil Development Company.

In a process for catalytically cracking hydrocarbon oils in the presence of a steam-sensitive catalyst, such as a synthetic silica-alumina gel, wherein the catalyst is alternately subjected to cracking and regeneration conditions and is exposed to the action of steam to purge volatilizable hydrocarbons prior to the regeneration, the activity of the catalyst is maintained by introducing ammonia into the zone in which the catalyst is initially exposed to the steam. An amount of ammonia of at least 5 mol-percent of the water vapor present is used.

U.S.P. 2,413,161. Surface Active Agents. E. Zerner and W. Kaplan to Sun Chemical Corporation.

Cracked mineral-oil fractions boiling between 150-300° C. and containing not more than 10-35 percent of unsaturated hydrocarbons are condensed with benzene, toluene, or xylene in the presence of $AlCl_3$. The condensation product is sulfonated. The products obtained are useful as surface active agents and detergents, wetting agents, and the like. It is



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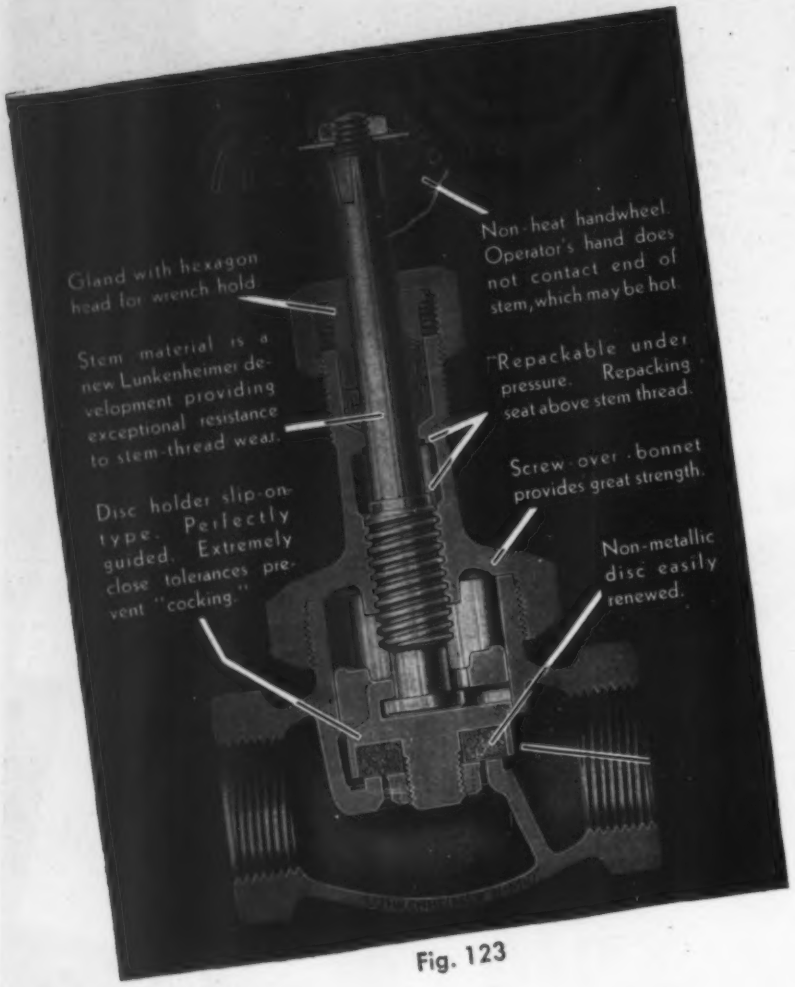
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Fig. 123

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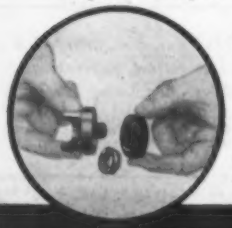

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unnecessary to separate the unsaturated components of cracked mineral oil before condensation.

U.S.P. 2,413,407. Method of Cracking Hydrocarbons in the Presence of a Diluent Gas. H. Dreyfus.

The cracking of hydrocarbons is effected in the presence of a diluent gas or vapor while the hydrocarbons are passing down a cracking chamber in the form of a self-supporting helical stream. At least part of the diluent is introduced at a point below the point of introduction of the hydrocarbons and in such a manner as to assume the form of a self-supporting helical stream merging with that of the descending hydrocarbons. Steam can be used as the diluent. The helical stream of hydrocarbons and diluent is obtained by spraying the components into the cracking chamber from tangentially arranged nozzles. High yields of unsaturated hydrocarbons and especially gaseous olefins are obtained.

ISOMERIZATION

U.S.P. 2,412,675. Isomerization of Saturated Hydrocarbons. J. D. Danforth to Universal Oil Products Company.

A liquid heated stream comprising a portion of isomerizable saturated hydrocarbon is passed through a bed of $AlCl_3$ to dissolve a portion of the $AlCl_3$. The resultant solution is introduced to a reaction zone containing a solid packing material. A second portion of the hydrocarbon is separately heated to a higher temperature and then directly introduced to the reaction zone. The amount and temperature of this second portion must be such as to maintain an isomerizing temperature in the reaction zone. $AlCl_3$ catalyst from said solution is deposited onto the packing material to an extent sufficient to maintain in the reaction zone an $AlCl_3$ concentration substantially greater than that of the solution. Isomerized hydrocarbon is recovered from the reaction products.

U.S.P. 2,413,691. Process for the Production of Neohexane Involving Catalytic Isomerization. C. C. Crawford, W. E. Ross, and S. H. McAllister to Shell Development Company.

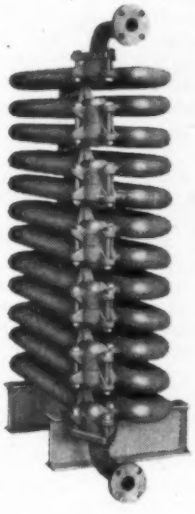
An open-chain hexane devoid of a quaternary C atom, such as methyl pentane or n-hexane, is isomerized in the absence of higher paraffin hydrocarbons and in the presence of an amount of 3-50 percent of a cycloparaffin hydrocarbon with 5-8 C atoms, such as methyl cyclopentane. The amount of the cycloparaffin shall be sufficient to inhibit degradation to isobutane. The $AlCl_3$ isomerization catalyst constitutes at least 10 percent by volume of the mixture and contains free $AlCl_3$ and 2-10 percent HCl. An isomerization temperature of 65-120° C. and a contact time of less than 40 minutes is used to effect conversion of at least 15 percent of the hexane to neohexane, which is separated from the reaction product by distillation.

ALKYLATION

U.S.P. 2,412,595. Process for Reaction of Aromatic Hydrocarbons with Normally Gaseous Unsaturated Hydrocarbons. W. N. Axe to Phillips Petroleum Company.

Aromatic hydrocarbons and normally gaseous unsaturated hydrocarbons are

SIMPLE *but* EFFECTIVE



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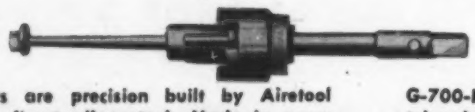

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contacted simultaneously with a catalyst comprising the addition compound of BF_3 with H_3PO_4. In addition to alkylation products, a gaseous mixture comprising unreacted unsaturated hydrocarbons and minor amounts of BF_3 is removed from the reaction zone. This gaseous mixture is scrubbed with H_3PO_4 to remove the BF_3 therefrom. BF_3 is added to the thus formed addition compound up to saturation. The resulting material is used as the catalyst in the contacting step. Benzene is, for example, alkylated by this process with ethylene. The yield of mono-alkylated products to be obtained by the use of the indicated catalyst may closely approach theoretical amounts.

U.S.P. 2,412,645. Treating Hydrocarbon Fluids. J. C. Munday to Standard Oil Development Company.

A catalytically cracked naphtha is separated into a light naphtha fraction of about 60-165° F. boiling range, an intermediate naphtha fraction of about 170-220° F. boiling range, and a heavy naphtha fraction of about 220-410° F. boiling range. The light fraction is alkylated with isobutane and the heavy fraction is catalytically cracked to higher-aviation-quality constituents. The alkylated products obtained are combined with the catalytically cracked heavy fraction to produce aviation gasoline. The intermediate naphtha fraction is discarded.

U.S.P. 2,412,726. Fluorine Removal Process. F. E. Frey to Phillips Petroleum Company.

In the alkylation of a low-boiling paraffin hydrocarbon, such as isobutane, with an alkylating reactant, such as butylene, in the presence of a HF catalyst, a liquid-hydrocarbon material comprising unreacted paraffin hydrocarbons and alkylate and dissolved HF together with organic fluorine-containing compounds is separated from the alkylation effluent. This material is subjected to condition such that substantially all of the HF and a substantial portion of the unreacted paraffin hydrocarbons are vaporized and substantially all of the alkylate together with high-boiling organic fluorine compounds remain in the liquid state. The resulting unvaporized liquid is subjected to dehydrofluorination conditions to convert the organically bound fluorine to HF. Effluent of the dehydrofluorination is fractionally distilled. The vapors from the vaporization step are condensed and cooled into a liquid-hydrocarbon phase and an undissolved liquid-HF phase. The liquid-hydrocarbon phase is passed to the fractional distillation as a reflux stream. A low-boiling fraction comprising free HF and a high-boiling fraction comprising unreacted hydrocarbons and alkylate free from HF are recovered from the fractional distillation.

U.S.P. 2,412,863. Circulation and Contacting of Acid Catalysts. J. C. Bolinger and P. W. Prutzman to Socony-Vacuum Oil Company.

A feed stream of mixed hydrocarbons to be subjected to catalysis in the presence of a liquid acid catalyst, as e.g., in the alkylation of isobutane with butene by means of HF or certain highly acid halides, is passed through a succession of contact zones containing bodies of the catalyst. The hydrocarbon stream enters the lower portion of each zone at velocities sufficient to insure turbulence

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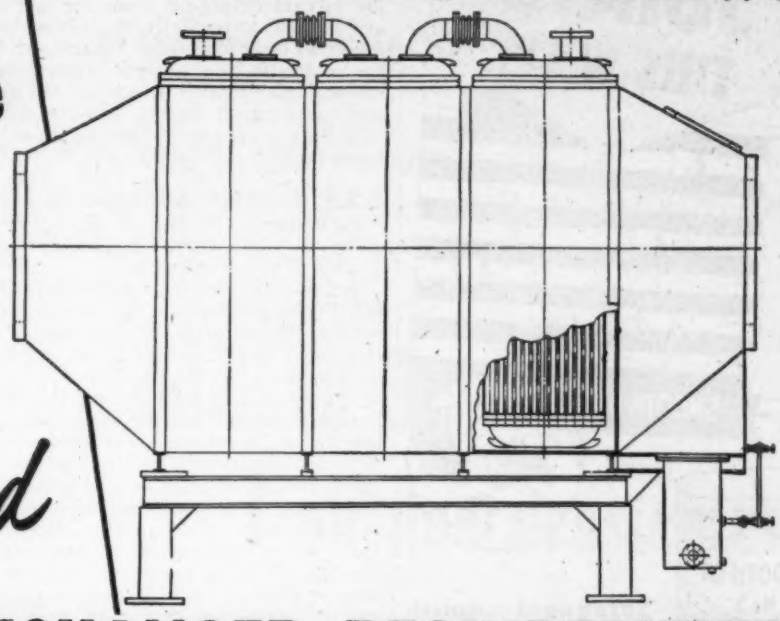

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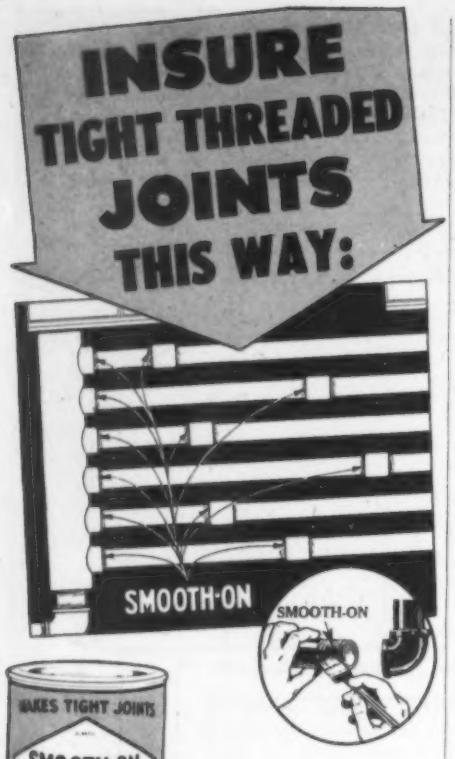

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therein and to carry a portion of the catalyst entrained in the hydrocarbons from zone to zone and out of the last zone. Enough catalyst is thus exchanged to permit maintenance of the catalytic efficiency of the bodies of catalyst through replacement of spent catalyst. The entrained catalyst is separated from the stream emerging from the last zone and is at least partially regenerated. Unreacted hydrocarbons separated from the catalyst-free stream are returned under high pressure to one of the zones, and regenerated catalyst is aspirated into this high-pressure stream of hydrocarbons.

U.S.P. 2,413,105. Alkylation of Hydrocarbons. G. B. Hatch, E. F. Pevere, L. A. Clarke, and F. H. Bruner to The Texas Company.

An isoparaffin is continuously alkylated with an olefin in the presence of an immiscible catalyst of greater density than that of the hydrocarbon employed, such as H_2SO_4 of 90-100 percent strength, by continuously introducing the hydrocarbons into a closed system containing circulating emulsion of mixed catalyst and hydrocarbons. A portion of this emulsion is continuously diverted to a separating zone where catalyst phase is settled from hydrocarbon phase and from which both phases are continuously discharged. Removal of the catalyst from the separating zone in accomplished as rapidly and completely as possible in order to reduce to a minimum the time of contact between catalyst and hydrocarbon after separation from the emulsion. At least a portion of the separated catalyst phase is recycled to the alkylation reaction zone.

U.S.P. 2,413,205. Dehydration of Hydrohalogen Acid by Distillation. J. C. Word, Jr., and J. J. Sims to Phillips Petroleum Company.

An essentially aqueous solution of hydrogen halide, particularly a HF solution as obtained in the alkylation of hydrocarbons by means of a HF catalyst, is concentrated by first adding ethylene glycol which forms a complex with the hydrogen halide. The liquid obtained is heated to a temperature above the boiling point of H_2O but below the temperature at which substantial dissociation of the complex ocurs, to drive out water. The partially water-freed residue is then distilled at a temperature within the dissociation range of the complex, e.g., at 325-385° F. Hydrogen halide is obtained in a concentrated form as distillate and ethylene glycol as residue. HF of about 60 percent concentration is, e.g., obtained and can be further concentrated by distillation to at least 90 percent.

U.S.P. 2,413,384. Production of Branched-Chain Paraffinic Hydrocarbons. L. Schmerling to Universal Oil Products Company.

Branched paraffinic hydrocarbons with at least 6 C atoms are obtained by reacting an alkyl halide containing at least 6 C atoms with an isoparaffinic hydrocarbon of not more than 5C atoms in the presence of a metal-halide catalyst of the Friedel-Crafts type. The amount of this catalyst shall be less than the stoichiometric amount required to form a double compound with the alkyl halide but sufficient to effect replacement of the halogen content of the alkyl halide with hydrogen. Paraffinic reaction products comprising said branched-chain paraffins are recovered.

U.S.P. 2,413,759. Alkylation of Paraffins. F. E. Frey to Phillips Petroleum Company.

An alkylatable paraffin hydrocarbon, such as isobutane, is inter-reacted with an alkyl halide, such as isopropyl chloride, in the presence of concentrated H_2SO_4 containing 0.5-5 percent by weight of aluminum sulfate. Paraffin hydrocarbons of higher mol weight, such as isoheptanes, are formed.

U.S.P. 2,413,777. Hydrocarbon Conversion. E. H. Oakley and L. F. Brooke to California Research Corporation.

Isoparaffinic hydrocarbons are alkylated with olefinic hydrocarbons by means of a catalyst comprising concentrated H_2SO_4 and an alkyl phosphoric acid in an amount sufficient to materially alter the catalytic activity of H_2SO_4 in the direction of producing alkylate of higher octane number. Monobutyl pyrophosphoric acid in an amount of less than 25 percent by weight of the catalyst is, for example, employed in the production of high anti-knock branched-chain paraffinic hydrocarbons boiling within the gasoline boiling range from lower-boiling hydrocarbons.

POLYMERIZATION, CONVERSION

U.S.P. 2,412,762. Manuacture of Butadiene from Propene and Formaldehyde. A. R. Workman to Cities Service Oil Company.

Butadiene is obtained by reacting propene with formaldehyde under anhydrous conditions at a temperature of 75-115° F. in contact with a catalyst mixture including zirconium chloride and zirconium dioxide. The zirconium chloride effects condensation of the reactants, and the zirconium dioxide converts the resulting condensation product to butadiene by dehydration.

U.S.P. 2,412,917. Method for Catalytic Conversion. T. P. Simpson, J. W. Payne and J. A. Crowley to Socony-Vacuum Oil Company, Inc.

A hydrocarbon-oil vapor is contacted at conversion temperature with a compact downwardly moving mass of particle-form, clay-type, solid catalytic material. The vapors are introduced at a flow rate not in excess of that sufficient to produce about 7-10 inches of water pressure drop per foot of vapor path through said mass. The conversion product is separated from this mass. The separated catalyst is moved through a regeneration zone as a compact moving mass while oxygen-containing gas is supplied thereto to burn contaminant deposits therefrom. Sufficient heat is removed to prevent the temperature of regeneration from rising above the point of damage for the contact mass. The hot regenerated mass is returned to the conversion zone.

U.S.P. 2,412,983. Process for the Conversion of Heavy Hydrocarbon Liquids. E. Hene, London.

A heavy hydrocarbon liquid having a boiling point of at least 350° C. and containing a substantial proportion of unsaturated compounds is distributed over a molten surface at a temperature of 430-500° C. at a controlled rate of feed such that each particle of the liquid is rapidly raised to a temperature at which it is partially vaporized and the

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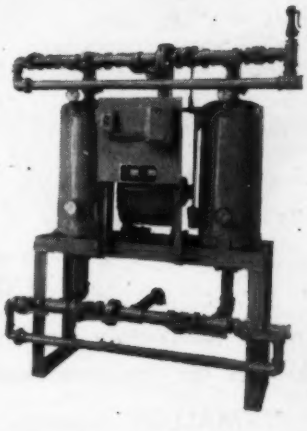

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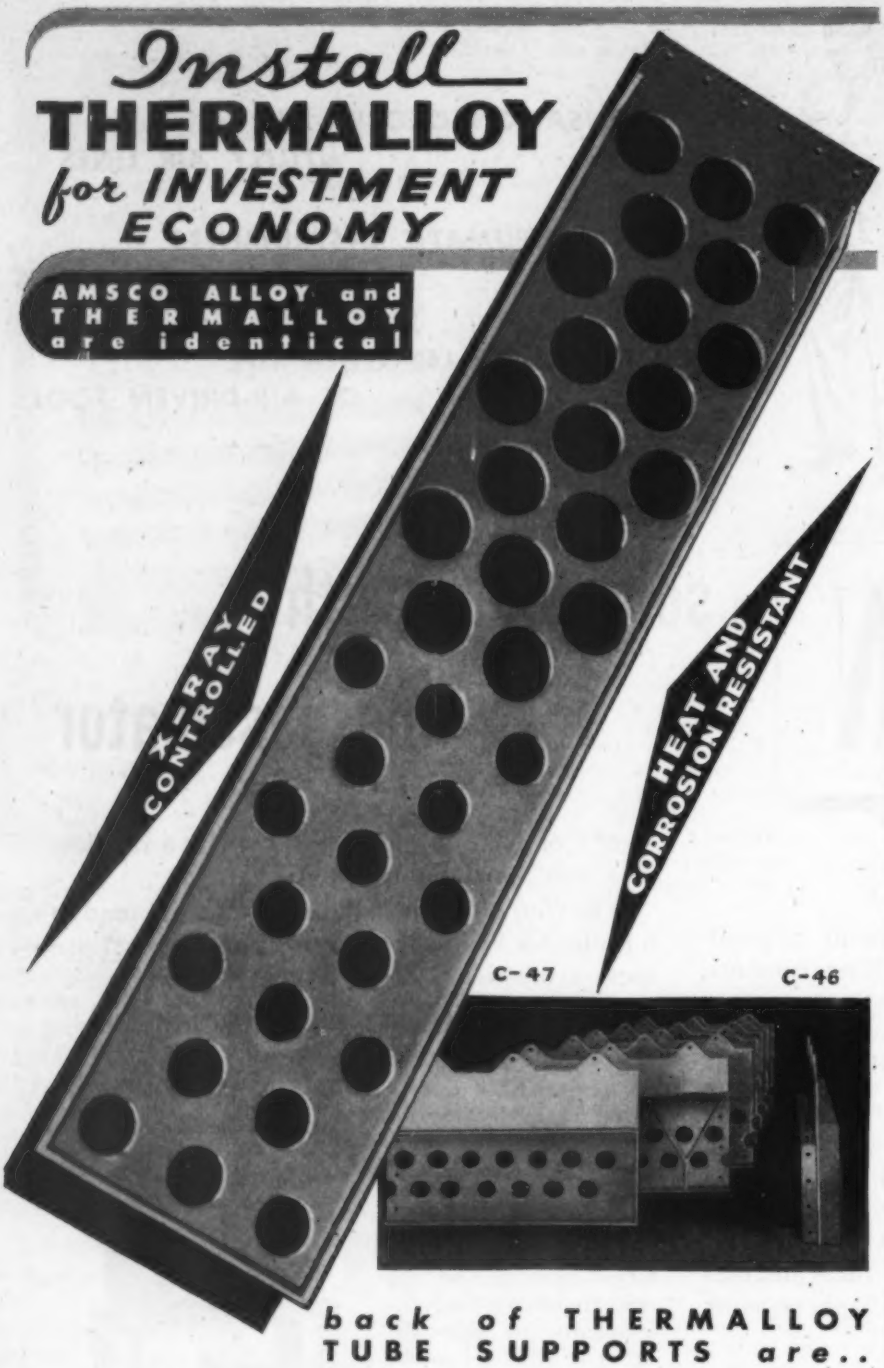

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unvaporized portion thereof remains in liquid condition. The resultant vaporous and liquid conversion products are immediately removed from the reaction zone before any substantial coking of the liquid occurs. The heavy hydrocarbon liquid yields a resinous material in the liquid conversion products which are subjected to solvent extraction to separate this resinous material. Valuable hydrocarbons of high molecular weight and mainly of a more saturated nature than the starting material are obtained by the process. Lead is, for example, used for preparing the molten surface.

U.S.P. 2,413,271. Catalytic Treatment of Hydrocarbons. H. R. Warrick to The Texas Company.

Hydrocarbon vapors are continuously charged into the lower portion of one of a plurality of reactors and passed at conversion temperature upwardly through this reactor in contact with comminuted catalyst held in suspension in the vapors. The catalyst is retained in the reactor in suspension in the current of upwardly flowing vapors while the vapors are discharged overhead. Used catalyst is simultaneously reactivated in a second reactor by establishing and maintaining a cyclic passage of the used catalyst through this reactor and an associated chamber which can be used alternatively with the plurality of reactors. The used catalyst is kept in suspension in a current of oxidizing gas. Resultant combustion gases are withdrawn while maintaining catalyst in cyclic passage. Processing in the first reactor and reactivation in the second reactor and associated chamber are then discontinued, the charge of hydrocarbon vapors is diverted to the second reactor and processed therein in the manner indicated before while the used catalyst is reactivated in the first reactor and the associated chamber.

U.S.P. 2,413,893. Copolymerization of Indenes and Piperylene. F. J. Soday to The United Gas Improvement Company.

A mixture containing piperylene and indene is contacted with 0.1-10 percent by weight of the total reactants of an acid-acting metallic halide catalyst at a temperature of —60 to 145° C. A benzene-soluble resinous copolymer of piperylene and indene is recovered which is compatible with drying oil. $AlCl_3$-diethyl ether can, for example, be used as the catalyst.

HYDROGENATION, DEHYDROGENATION, AROMATIZATION

U.S.P. 2,412,600. Catalysis of Hydrocarbons by Gel-Type Catalysts. R. E. Burk and E. C. Hughes to The Standard Oil Company (Ohio).

A hydrocarbon of at least 6 C atoms or a naphtha is contacted at elevated temperature with a mass consisting of Al, Cr, and Mo gel type oxides coprecipitated from metal salts with a hydroxide precipitant. Dehydrogenation and cyclization of hydrocarbons can be effected by such a catalyst, using a pressure of 50-300 psi and a temperature of 850-1200° F. A suitable catalyst is, e.g., formed by treating an Al salt and a Cr salt with a hydroxide precipitant in the presence of ammonium para-molybdate in proportions to provide 10 percent

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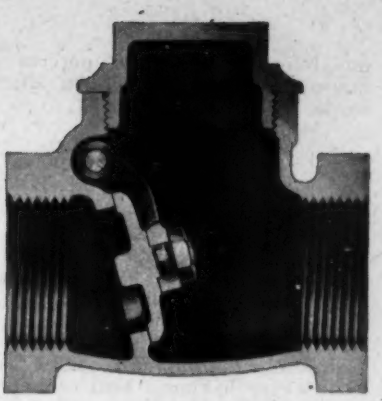

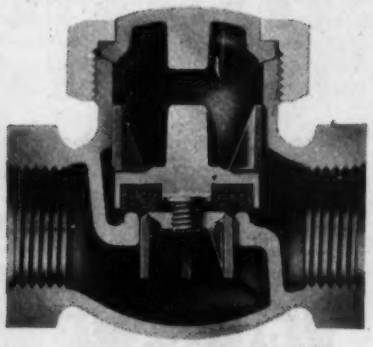

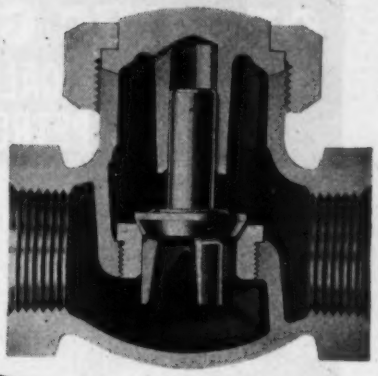

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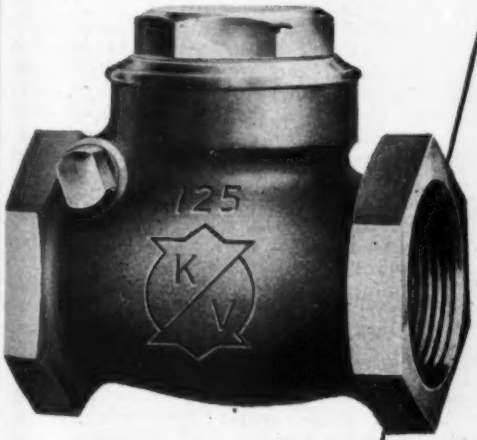

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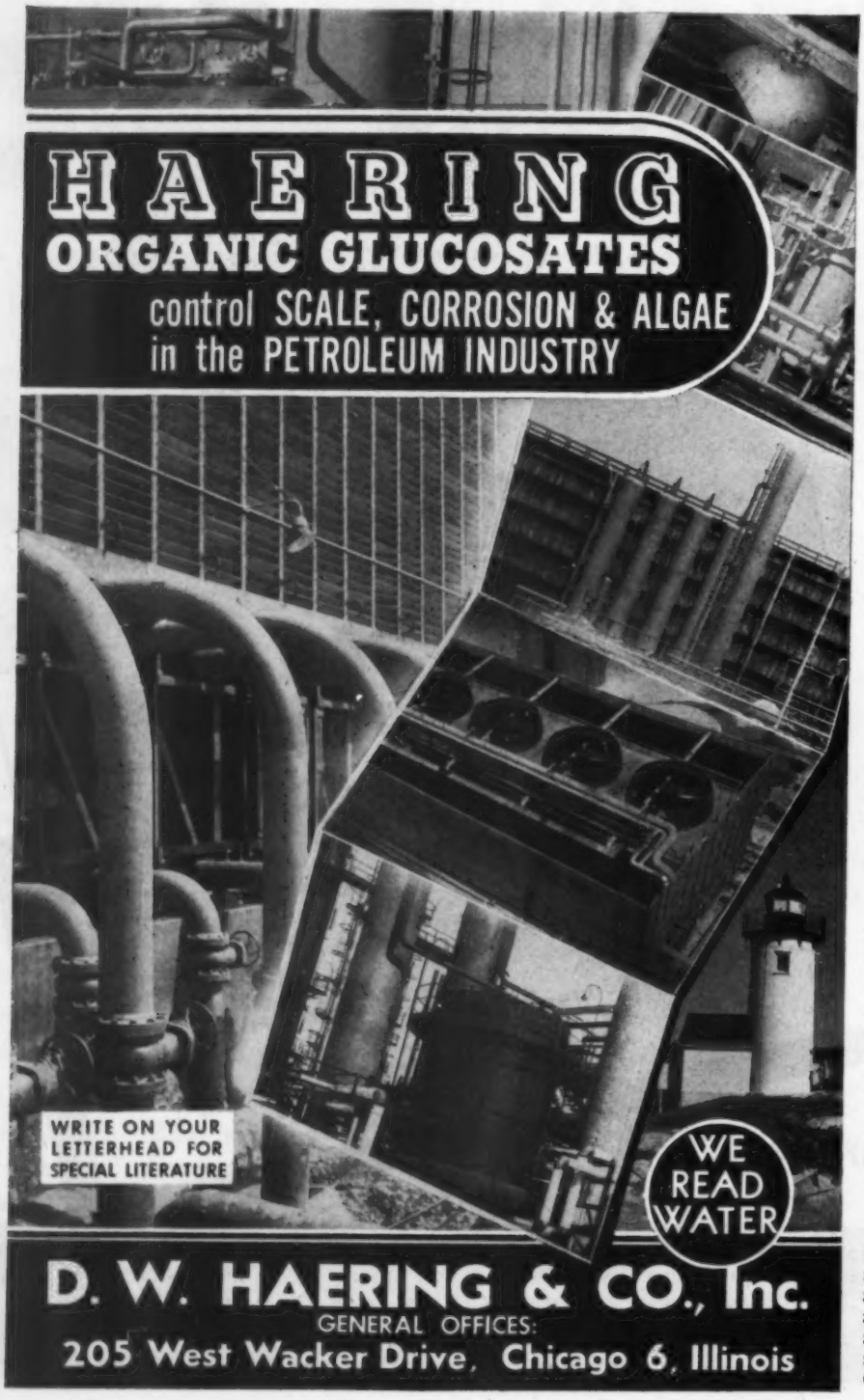
U.S.P. 2,412,828. Preparation of Aromatic Hydrocarbons from Hydrocarbon Mixtures. E. A. Naragon to The Texas Company.

A naphtha feed hydrocarbon mixture boiling above 175° F. and containing 10-80 percent by volume of aromatics is contacted, preferably at a temperature of 60-120° F., with an aluminum halide hydrocarbon complex of the Friedel-Crafts type having a heat of hydrolysis of 290-330 cal. per gram of complex. A primary extract phase comprising complex, aromatic and some non-aromatic hydrocarbons, and a primary raffinate phase consisting essentially of non-aromatic hydrocarbons are formed. The raffinate phase is discharged. The extract phase is subjected to countercurrent contact with substantially less than an equal volume of a wash solvent consisting of saturated petroleum hydrocarbons at a temperature in the range of 60-120° F. so that non-aromatic extract hydrocarbons are displaced therefrom leaving a secondary extract phase comprising complex, aromatic primary extract hydrocarbons, and a small amount of wash solvent. The displaced hydrocarbons are discharged. The secondary extract phase is countercurrently washed with a further quantity of the wash solvent in a proportion of from 1-3 volumes of solvent per volume of secondary extract under similar conditions so as to displace the aromatic extract hydrocarbons from the complex.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U.S.P. 2,412,911. Butadiene Manufacture. H. W. Scheeline to Standard Oil Development Company.

A petroleum fraction consisting mainly of butene-2 is catalytically dehydrogenated to form butadiene. Hydrocarbons with 3 or less C atoms and 5 or more C atoms are separated from the reaction mixture obtained. The residual C_4 hydrocarbons are mixed with a fraction of hydrocarbons with 4 C atoms obtained by cracking a petroleum oil. This mixture is extractively distilled in the presence of a solvent having a preferential solvent action for butadiene and butene-2 and reducing the volatility of these two compounds with respect to the other hydrocarbons present. The solvent then is separated from the dissolved mixture of butadiene and butene-2 which are separated by fractionation. The butene-2 is recycled to the dehydrogenation unit.

U.S.P. 2,412,936. Production of Cyclopentene. H. J. Hepp to Phillips Petroleum Company.

A mixture of cyclopentane and hydrogen containing at least 25 and less than about 80 mol percent of cyclopentane is contacted with a dehydrogenation catalyst, such as a chromium oxide-alumina catalyst, at a temperature of 1000-1300° F. for a sufficient period of time to convert at least 20 percent of the cyclopentane to unsaturated cyclic hydrocarbons containing a 5-C-atom ring, the major portion of which consists of cyclopentene.

DESULFURIZATION

U.S.P. 2,413,312. Catalytic Finishing of Gasolines. R. M. Cole to Shell Development Company.

Sulfur-containing olefinic gasoline is treated in the presence of an excess of hydrogen and a sulf-active hydrogenation catalyst, such as an oxide or sulfide of V, Cr, Mn, Fe, Co, Ni, Mo, or W, at a temperature of 400-825° F. under conditions such that only a partial desulfurization is effected and only a minor part of the olefins is hydrogenated. The partially desulfurized material is separated into a lower-boiling and a higher-boiling olefinic fraction. The higher-boiling fraction is then treated in the presence of an excess of hydrogen and a sulf-active hydrogenation-dehydrogenation catalyst, such as a nickel sulfide-tungsten sulfide catalyst, at a temperature of 850-1000° F. under conditions such that further desulfurization and substantial saturation of olefins is effected. The product of this second treatment is blended with the lower-boiling fraction from the first treatment. A refined olefinic gasoline with an octane number at least as high as that of the starting material is obtained.

HEAVY OILS AND WAXES

U.S.P. 2,412,557. Lubricating Oils. C. M. Blair, Jr., to Petrolite Corporation, Ltd.

The lubricating composition described in this patent comprises a lubricating oil and 0.25-5 percent of an unsaturated amine-alpha-beta unsaturated carboxylic acid addition-condensation polymer. The amine contains at least one ethylenic unsaturation, has at least 4 singly-bound C atoms between the ethylenic double

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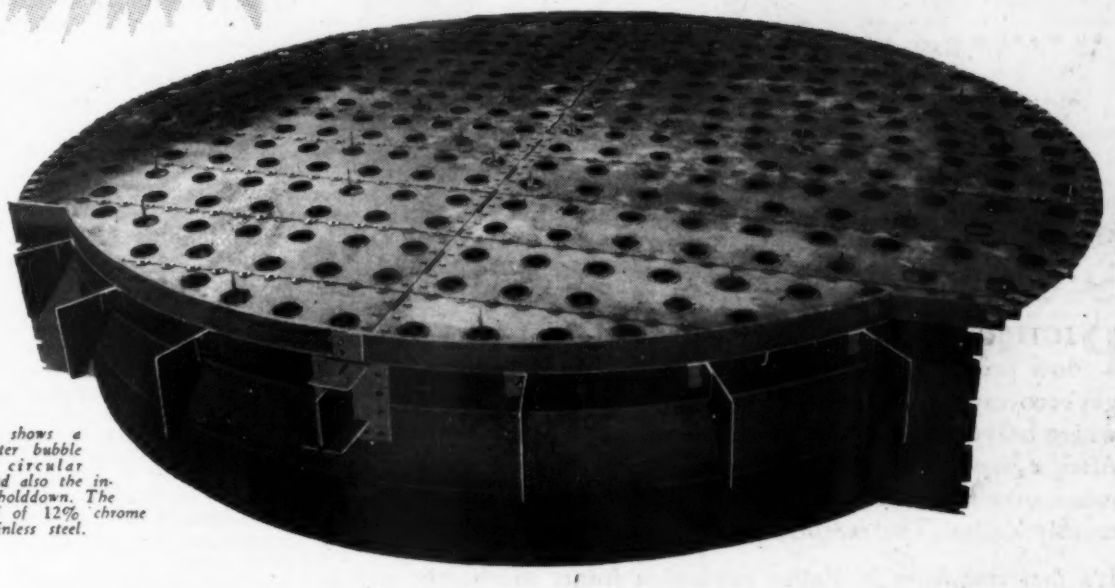

This picture shows a 16'-4" diameter bubble tray with circular downcomer and also the individual cap holddown. The tray is made of 12% chrome type 410 stainless steel.

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MANUFACTURED UNDER U. S. PATENT NO. 2,210,808; NO. 2,309,309; NO. 2,338,926; NO. 2,341,091 AND OTHER PATENTS PENDING.

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bond and the nearest amine nitrogen atom, has at least one hydrogen atom directly linked to an amino nitrogen atom, and contains less than 32 C atoms per nitrogen-attached hydrocarbon group. The acid contains less than 10 C atoms and is free from vinyl radicals. The additive used can, for example, be formed by reacting maleic anhydride with oleyl amine and heating the reaction product until a cryoscopic mol weight of 2400 is obtained. The viscosity index of the lubricant is increased by the addition.

U.S.P. 2,412,633; 2,412,634. Lubricants. M. L. Schwartz to Standard Oil Company (Indiana).

The lubricating oil according to U.S. P. 2,412,633 contains 1-10 percent of castor oil, castor amide, or glycerol monoricinoleate, 0.1-2 percent of sulfurized terpene, and corrosion inhibiting, small amounts of a soap of a preferentially oil-soluble petroleum sulfonic acid.

According to U.S.P. 2,412,634, an amount of 5.40 percent of said soap and 0.5-10 percent of one of said castor compounds is used without the addition of sulfurized terpene.

U.S.P. 2,412,708. Lubricating Oils. C. M. Blair, Jr., to Petrolite Corporation, Ltd.

The lubricating oil contains 0.025-5 percent of an alpha-beta, unsaturated carboxylic acid amine mixture addition-condensation polymer which is the reaction product of (a) an unsaturated amine with at least one amino hydrogen atom, at least one ethylenic unsaturation, and at least 4 singly-bonded C atoms between the ethylenic double bond and the nearest amino nitrogen atom, and contains less than 32 C atoms per nitrogen-attached hydrogen group, in admixture with a saturated amine containing at least 1 amino hydrogen atom, free from aryl radicals and also containing less than 32 C atoms per nitrogen-attached hydrocarbon group; and (b) an alpha-beta, unsaturated, carboxylic acid containing less than 10 C atoms and free from vynil radicals. (See the additive used in U.S.P. 2,412,557.)

U.S.P. 2,412,903. Compounded Lubricating Oil. R. J. Miller and J. T. Rutherford to California Research Corporation.

The lubricant according to this patent comprises a major portion of hydrocarbon lubricating oil, 0.1-5 percent by weight based on the compounded lubricant of an oil-soluble alkaline earth metal phenate, such as a calcium salt of a phenol alkylated with a butene polymer averaging about 16 C atoms per molecule and boiling between 375 and 500° F., and 0.1-5 percent of an oil-soluble zinc thiocarbamate, such as a zinc dibutyl dithiocarbamate.

U.S.P. 2,412,919. Lubricant. L. G. Vande Bogart and R. W. Manuel to Crane Company.

The grease-like lubricant described comprises 5-45 percent of castor oil and 5-45 percent of petroleum oil. The proportions and character of these oils are such that they are not mutually soluble. Bentonite clay and non-aqueous and soap-free liquid stabilizing agent consisting of the lower alkyl phosphate group, such as triethyl and tributyl phosphate, are admixed to the oil mixture. The amount of the oils present in the compound is greater than that represented by the oil-absorption value of the

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bentonite clay. The amount of the stabilizing agent is sufficient to prevent a separation of the bentonite clay and the oils into two distinct solid and liquid phases. For example, 0.5-5 percent by weight of the stabilizing agent is employed. The lubricant has high load-carrying properties.

U.S.P. 2,412,956. Lubricant Containing Alkyl Piperidine Salts. G. E. Barker to Elgin National Watch Company.

This lubricant consists of an organic lubricant liquid, such as a hydrocarbon oil, in which 0.05-1.0 percent of an alkyl-piperidine salt of a fatty acid is dissolved. The alkyl group and the acid group of this combination have 5-30 C atoms, each. Dodecylpiperidine stearate is, for example, used as the additive.

U.S.P. 2,413,188. Lubricants, etc. J. M. Musselman and H. P. Lankelma to The Standard Oil Company (Ohio).

The lubricant consists of a mineral lubricating oil containing about 0.1-10 percent of a salt of an alkyl sulfuric acid with at least 6 C atoms, such as aluminum octyl sulfate, ammonium hexyl sulfate, or sodium lauryl sulfate. The lubricant is capable of maintaining desirable body and of cleaning metal surfaces under heat and drastic usage.

U.S.P. 2,413,311. Lubricant and Process for Preparing Same. C. A. Cohen to Standard Oil Development Company.

A finely divided siliceous filter aid, such as diatomaceous earth, tripoli, or finely ground silica, is contacted with an acid-treated oil containing pre-formed pepper sludge in dispersion, dissolved SO_2, and oil-soluble sulfonic acids having a combining weight of at least 400. Pepper sludge and filter aid are co-precipitated. SO_2 is removed from the oil, and the oil-soluble sulfonic acids then are neutralized with a basic compound of polyvalent metal to form soluble sulfonates in the acid-treated oil. The removal of the dissolved SO_2 may be carried out by air blowing or under reduced pressure. Oxides, hydroxides, or carbonates of alkaline earths can be employed for neutralizing the sulfonic acids. The sulfonic acids present in the process are formed by a pre-treatment of the hydrocarbon lubricant oil employed with H_2SO_4 of at least 100 percent concentration.

U.S.P. 2,413,332. Composition of Matter Suitable for Use as a Lubricant and Lubricant Comprising the Same. J. M. Musselman to The Standard Oil Company (Ohio).

The reaction product of a phosphorus sulfide and an oxygen-containing wax, in admixture with a plural metal compound of this reaction product is used as a lubricant or as an addition agent in an amount sufficient to improve the characteristics of lubricating oils and greases. A major portion of the oxygen in the wax is replaced by sulfur when the wax is treated with phosphorus sulfide. At least one of the metals used for forming the plural metal compound is preferably an alkali or an alkaline earth metal. This lubricant is useful for high pressures and high temperatures, e.g., in internal combustion engines.

U.S.P. 2,413,353. Cutting-Oil Composition. B. F. Hunter and H. P. Hobart to Gulf Oil Corporation.

This cutting-oil composition comprises at least 50 percent by weight of a light mineral oil boiling above 350° F.

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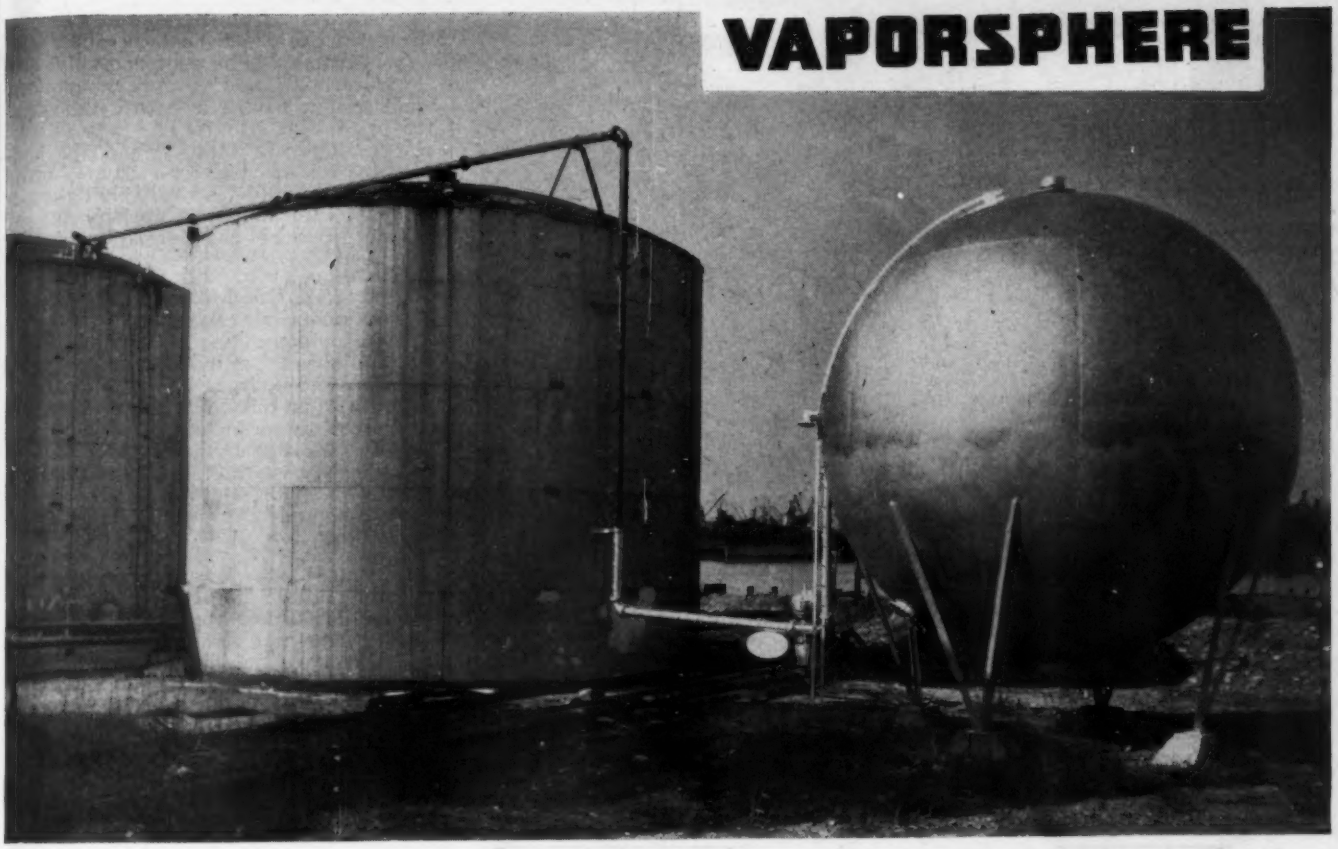
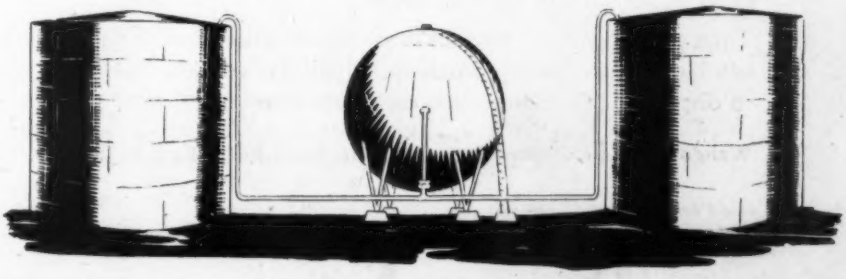


YOU have probably learned from experience that you cannot store volatile liquids in tanks with fixed roofs without having substantial evaporation loss due to *breathing* and *unbalanced filling and emptying*. You can, however, connect these same storage tanks to a Horton Vaporsphere and you will have an efficient evaporation-saving system.

The photo above shows the Vaporsphere used in this manner at a bulk plant in Alabama. It is connected to a 5,000-bbl. and a 6,000-bbl. umbrella-roof tank, both storing gasoline. The vapor spaces of the two tanks are connected to the Vaporsphere by large diameter piping. Vapor displaced by filling, or forced out of the tanks by expansion due to an increase in temperature, flows through the vapor line into the Vaporsphere. This vapor, which normally would be lost to the atmosphere, is stored temporarily in the Vaporsphere and returned to the tanks when the conditions which caused its displacement are reversed.

The Horton Vaporsphere may be connected to existing tanks without taking them out of service. Get complete details from our nearest office. Ask for Bulletin D.

In the above photo the two flat-bottom tanks are located close together and, therefore, the piping to the Vaporsphere passes over one of the tanks. Ordinarily the vapor lines are placed close to the ground. The sketch at the right shows a typical arrangement of two cone roof tanks and a Horton Vaporsphere.



CHICAGO BRIDGE & IRON COMPANY

***Plants in* BIRMINGHAM, CHICAGO and GREENVILLE, PENNSYLVANIA**

Atlanta 3	2146 Healey Building	New York 6	3302—165 Broadway Building	Philadelphia 3	1608—1700 Walnut St. Building
Birmingham 1	1548 North Fiftieth Street	Cleveland 15	2234 Guildhall Building	Los Angeles 14	1434 Wm. Fox Building
Houston 1	5626 Clinton Drive	Chicago 4	2463 McCormick Building	Havana	402 Abreu Building
Tulsa 3	1626 Hunt Building	San Francisco 11	1251-22 Battery St. Building	Detroit 26	1520 Lafayette Building

REPRESENTATIVES AND LICENSEES

In France—Ateliers et Chantiers de la Seine Maritime, Paris
In France—Constructions Metalliques de Provence, Arles-sur-Rhone
In Venezuela—Chicago Bridge & Iron Company, Limited, Apartado 1348, Caracas
In Argentina—Leopoldo Sol & Cia., Reconquista 558, Buenos Aires
In Great Britain—Whessoe, Limited, Darlington, England
In Great Britain—Motherwell Bridge & Engineering Company, Limited, Motherwell, Scotland
In Canada—Horton Steel Works, Limited, Fort Erie, Ontario

"Fire protection here calls for instant detection—and prompt extinguishing"

Fire in a plant powerhouse *could* result in a lengthy shutdown —and the need of costly power purchases from outside sources.

Let a Kidde representative show you how this can be avoided!

When *Kidde* Systems, Wheeled Units and Portables protect your electrical equipment, fires are quickly and safely extinguished. *Kidde* equipment discharges *only* clean, non-conducting carbon dioxide—and discharges it entirely by its own stored energy.

No danger of short circuits or flashovers. No corrosion of metal parts. . . . no risk of damage to insulation. No danger of the extinguishing equipment failing because pumps or other power sources are shut down!

There may be *other* fire hazards in your plant where the advice of a Kidde representative would be helpful. He's always ready to discuss your fire protection problems with you.

Walter Kidde & Company, Inc., 348 Main Street, Belleville 9, N. J.

The word "Kidde" and the Kidde seal are trade-marks of Walter Kidde & Company, Inc.

Kidde

FIRE PROTECTION HEADQUARTERS

and having a viscosity not greater than 40 SUV at 100° F., about 1-20 percent of a mineral oil with a viscosity between 100 and 200 SUV at 210° F., about 0.5-15 percent of a fixed fatty oil, about 0.03-5 percent of a petroleum oil antioxidant, and up to 10 percent of the residue which remains after the removal of the solvent from the extract obtained by extracting a petroleum distillate with a solvent capable of removing polycyclic hydrocarbons and sulfur compounds, said residue having a viscosity of about 55-70 SUV at 210° F. The fatty oil can, for example, be lard oil and the antioxidant can be tri-(tertiary-amylphenyl)-phosphite. A relatively light cutting oil with improved lubricity and extreme pressure characteristics is thus obtained.

U.S.P. 2,413,718. Lubricant. B. H. Lincoln and G. D. Byrkit to Continental Oil Company.

The lubricant comprises a major proportion of a hydrocarbon oil and .001-20 percent of an oil-soluble halogen-bearing ester containing at least 1 carbon-boron bond. Chlorophenyl-boric acid dibutyl ester is, for example, used for this purpose. The lubricant has improved film strength and extreme pressure characteristics.

U.S.P. 2,413,852. Rust-Inhibiting Lubricant. W. R. Turner to The Atlantic Refining Company.

The lubricant comprises hydrocarbon oil and a rust-inhibiting quantity of an oil-soluble reaction product of an alkyl acid phosphate with 8-16 C atoms in the alkyl group and a branched-chain alkylamine with 4-16 C atoms in the alkyl group. Between 0.005 and 1.0 percent by weight of the reaction product of lauryl acid phosphate and di-2-ethyl hexylamine, for example, is employed. An amine antioxidant, such as 0.05-0.5 percent of phenyl alpha naphthylamine, can be added.

U.S.P. 2,413,972. Lubricant. R. D. Herlocker, M. P. Kleinholz, and F. M. Watkins to Sinclair Refining Company.

The patent relates to a turbine oil which comprises a petroleum lubricating oil containing a minor proportion of tris-(morpholinomethyl)-phenol or bis-(morpholinomethyl)-phenol in sufficient amount (0.2-1.0 percent) to retard rusting of metal parts.

Celanese Corporation Sets Up Fellowship at Michigan

A Celanese Corporation fellowship in chemical engineering at the University of Michigan has been announced by Celanese Corporation of America. The fellowship is for a term of five years from the time of the appointment of the first recipient, and the subjects to be investigated under the fellowship shall relate to the field of plastics and high polymers.

A Celanese Corporation fellowship in the general field of chemical engineering was established at Princeton University in June, 1945.

Mason Promoted

H. A. Mason, until recently superintendent of the laboratories at General Petroleum Corporation's Torrance refinery, has been promoted to the position of assistant to the manager of laboratories, with headquarters in the home office of the company at Los Angeles.

NEW EQUIPMENT

. . . MANUFACTURERS' · LITERATURE . . .

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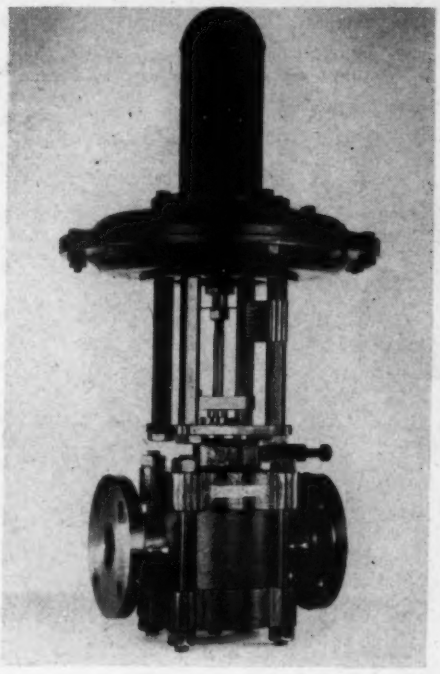
1—Diaphragm Motor Valve

Fischer & Porter Company, Hatboro, Pennsylvania, has announced a new design in diaphragm motor valve construction. These valves have been designated "The Valvrator Line," and are an outgrowth of the recording-controlling flow-rate meter which heretofore has been the principal product of this manufacturer.

Design features include dished-bonnet construction with flush-seated bodies and through-bolts. No bonnet flanges are included in the body construction, and the design results in a compact unit, with simplified cleanout characteristics, and the possibility for fabrication in nearly all corrosion-resisting materials. The valve is made in 150-pound construction in sizes from ¼- to 1-inch screwed, and 1- to 3-inch flanged.

Complete details are available in Catalog Section 70-A, available by writing.

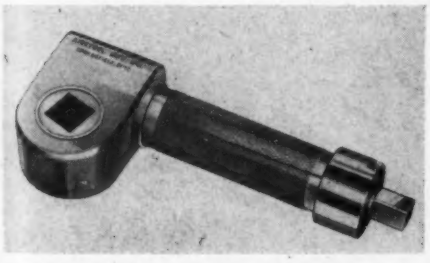
CHECK THE CARD AT PAGE 220



Fischer & Porter Diaphragm Motor Valve.

2—Right-Angle Drive

The Airetool Manufacturing Company, 300 South Center Street, Springfield, Ohio, announces production has begun on a new improved right-angle gear drive for tube expanders used through small headers and in hard-to-get-at places. The gear drive's one-piece housing is machined from solid bar steel. It has fewer movable parts than



Airetool Right-Angle Gear Drive

are normally found in this type tool. All moving parts are completely inclosed with one-shot lubrication that will last a longer than usual period. The grip is knurled for easier handling. The gear drive is made in sizes for ½-, ¾- and 1-inch square connections.

CHECK THE CARD AT PAGE 220

3—Steam Trap

Armstrong Machine Works, 852 Maple Street, Three Rivers, Michigan, has announced development of a new small all-steel steam trap for service on pressures up to 250 psi. The trap embodies the basic Armstrong inverted-bucket and free-floating-valve-lever design. The body and cap are steel forgings, capable of resisting considerably in excess of normal operating pressures. Internal operating mechanism, including the valve seat, is stainless steel. The trap measures 4¼ inches in diameter at the cap, with an overall height of 7 inches. Capacity averages 900 pounds of condensate per hour, continuous discharge.

The maker believes this new trap will have special appeal in oil refineries, chemical plants and other process industry plants where the trend is toward using all-steel pipe and fittings for pressure service. The forged-steel construction makes the trap resistant to impacts, and also retains its high strength characteristics when exposed to high temperatures, it was pointed out.

Armstrong Forged-Steel Steam Trap

CHECK THE CARD AT PAGE 220

4—Silicone Catalog

Dow Corning Corporation, Midland, Michigan, recently has issued the third edition of its catalog "Dow Corning Silicones, New Engineering Materials." It lists all silicone products now available, and contains many charts and graphs, descriptive of the unusual properties of this rapidly increasing family of organo-silicon-oxide polymers.

CHECK THE CARD AT PAGE 220

5—Liquid-Level Indicator

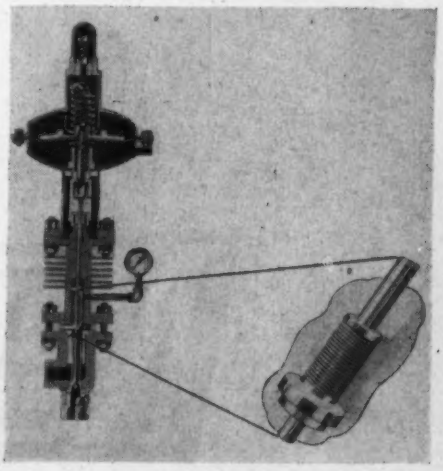
The Reliance Gauge Column Company, Cleveland, announces a newly styled, newly engineered model of the "Eye-Hye" remote-reading liquid-level indicator which it has marketed for 10 years. Besides giving the device a new, modern face plate, the construction has been altered to achieve greater compactness and certain structural advantages. Use of a plastic diffusing screen improves the distribution of the interior illumination. The case has been made drip-proof and the arrangement of the interior more accessible for cleaning and renewing of illuminating lamp. The new Model E-32 is the first of several which will replace the original series. It is described in Bulletin 452.

CHECK THE CARD AT PAGE 220

6—Packless Diaphragms Control Valve

Hammel-Dahl Company, 243 Richmond Street, Providence 3, Rhode Island, has announced elimination of stuffing boxes in its 3000 B Series of Packless Diaphragm Control Valves. Use of packless bellows sealed construction provides a leak-proof control valve and simplifies control of many difficult processes involving toxic fluids or in other service where leakage cannot be tolerated, it was said. The packless design can be applied to any size or type Hammel-Dahl Flo-tested Control Valve for a working pressure of 500 psi at any temperature between minus 70 to plus 750 degrees F.

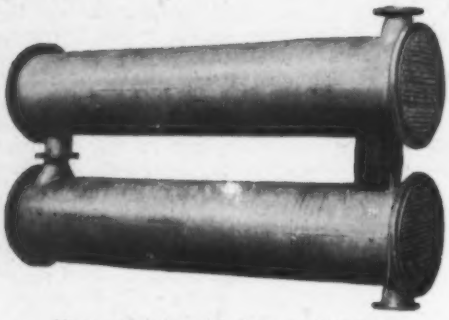
The bellows is housed in a unit assembly and replaces the conventional valve bonnet. The bellows unit is piloted into the valve body and retained between two stainless-steel gaskets by the bonnet assembly. It is protected from over-ranging by travel stops. It is properly supported internally and ex-



Hammel-Dahl Packless Diaphragm Control Valve.

NOOTER ENGINEERED FABRICATION MEANS SAFETY, STRENGTH and CORROSION-RESISTANCE

5'6"x120' steel fractionating tower, complete with bubble trays and caps — fabricated by Nooter.



Nooter — fabricated exchanger of Hastelloy throughout including tubes, shell, flanges, etc.

Jacketed stainless steel U-tube with polished inner surface — completely fabricated by Nooter.

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Reading blue prints and following your specifications are only a part of Nooter complete fabrication service to refiners and producers . . . Technical knowledge of pure metals and alloys, their properties and behaviors, —as well as experience in fabrication of petroleum processing equipment—are available to you at Nooter.

Tanks, vessels, towers and all their integrated parts are constructed to your specifications. A.P.I.—A.S.M.E. code welded construction.

WRITE FOR YOUR COPY OF THE NEW CORROSION—RESISTANT METAL TABLES—COMING OFF THE PRESS, SOON.

NOOTER ST. LOUIS

JOHN NOOTER Boiler Works Co. • 1404 S. Second St., St. Louis 4, Mo.

ternally without any possibility of the convolution rubbing any adjacent surface. A modified packing gland is employed as an emergency seal, should the bellows fail due to corrosion or as a result of mechanical fatigue. A telltale gauge can be provided to indicate positively a bellows failure.

The bellows unit can be inspected or replaced in the field. Damage to the bellows from torsional twisting in assembly or disassembly of the valve plug and stem is prevented by a hexagonal stem passing through the mounting ring.

The bellows unit is available in two-ply Type 347 stainless steel as standard. It may be mounted on any size screw-end valve from ½-inch to 1½-inch and flanged-end valve from 1¼-inch to 12-inch. Whole valve lift is assured in all sizes. This design can be provided with any type Hammel-Dahl valve such as Microflo, Venturiflo, Three-way or Internal Pilot Balanced Piston Type, as well as all standard constructions.

CHECK THE CARD AT PAGE 220

7—Fire Blanket

A new type fire blanket is being manufactured by United States Rubber Company.

The blanket can be used as an emergency wrap if a person's clothing is aflame; as an auxiliary fire extinguisher, to put out flames from vents, manholes, small containers or tanks; as a fire shield so that fire fighters can approach within effective range of liquid fires; as a welding screen against welding rays and sparks, and as a salvage blanket to protect materials from water, chemicals and heat.

It is 60 by 80 inches in size and made of high tensile strength glass cloth, impregnated on both sides with synthetic rubber. Hems and seams are vulcanized and folds are heat creased. It is equipped with large brass grommets at the top and a weather-resistant bracket so it can be hung up outdoors or indoors and easily removed or replaced. For quick identification, it is red in color and marked with the words, fire blanket.

Qualities claimed for the fire blanket are that it is flameproof, heat resistant, chemical resistant, water resistant, mildew proof, vermin proof, non-shrinkable, non-stretchable and unaffected by heat or cold.

CHECK THE CARD AT PAGE 220

8—Stainless-Steel Laboratory Filters

Micro Metallic Corporation, 193 Bradford Street, Brooklyn 7, New York, has announced production of stainless-steel laboratory filters with porous stainless-steel filter elements. Included are filter crucibles, Buchner funnels, gas-dispersion tubes and filter tubes.

These laboratory filters are made up by welding the porous stainless-steel filter element into carefully designed stainless filter bodies. Advantages include freedom from breakage by handling or thermal changes, and resistance to alkali and many acids.

Filters are available in five porosities.

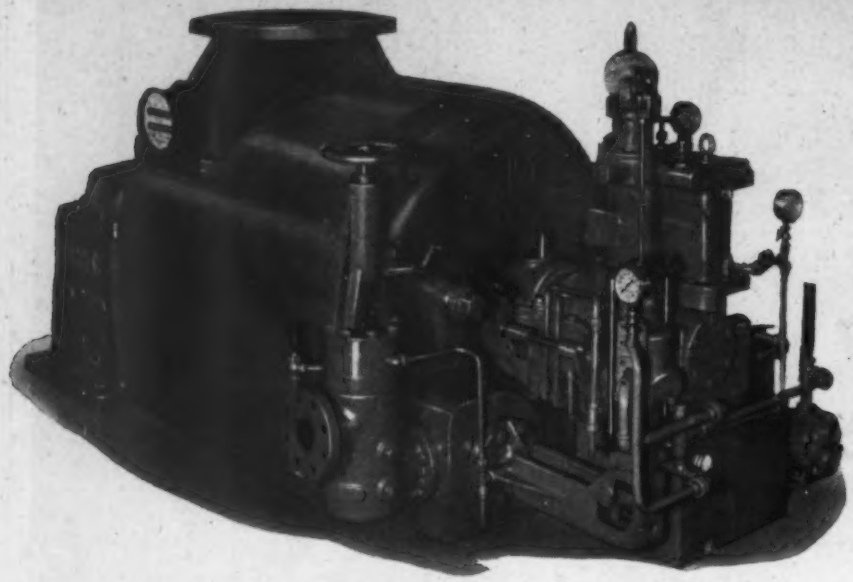
CHECK THE CARD AT PAGE 220

9—Tubing-Pipe Adapter

Electric Steel Foundry, 2141 NW 25th Avenue, Portland, Oregon, has announced an adapter which makes possible use of thin-walled stainless-steel tubing in piping installations where standard stainless pipe also is used.

This adapter fitting permits incorpora-

WYATT METAL & BOILER WORKS



One of two MURRAY multistage steam turbines, designed to deliver 685 horsepower on steam at 110 pounds and exhausting to 26½ inches Hg vacuum, furnished to drive high speed centrifugal compressors for refrigeration in one of the most prominent Southern Hotels. A third unit has recently been shipped. Equipment includes variable speed oil relay governor, trip and throttle valve, strainer surrounding the double seated balanced governor valve, and force feed lubrication.

MURRAY
MULTISTAGE STEAM TURBINES

★ ★ ★ ★

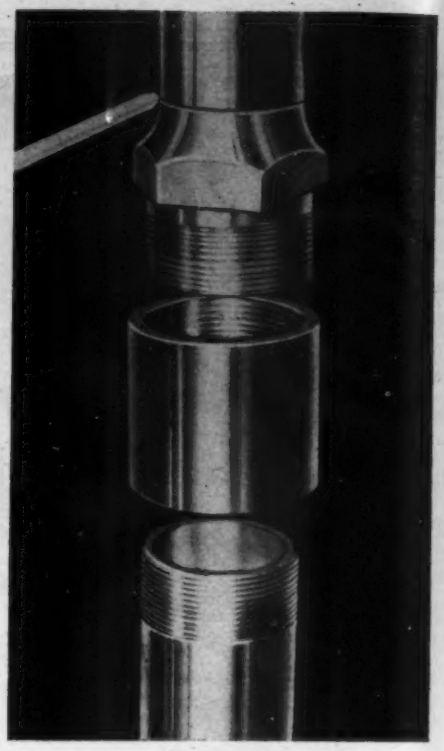
The complete MURRAY line of steam turbines includes all variations of mechanical drives, and turbine generator sets up to and including 3000 KW rating.

Sales Representatives for Murray products are located in all principal cities.

MURRAY IRON WORKS COMPANY

BURLINGTON, IOWA

Builders of Steam Power Equipment for Three Quarters of a Century



Esco P-T Adapter.

tion of stainless tubing in lighter take-off systems from an already established standard piping layout, and also makes practical use of tubing in place of pipe in making installations where tubing would be equally suitable in other respects, according to the manufacturer. It is called the Esco P-T Adapter.

The tubing is butt-welded to the adapter and thus is permanently fitted with standard pipe threads and can be assembled as any pipe. A system using this method is readily dismantled for cleaning or inspection.

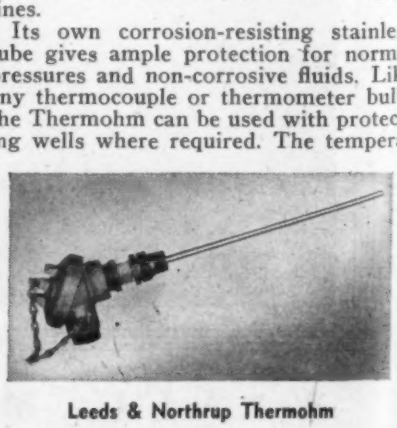
CHECK THE CARD AT PAGE 220

10—Fast-Acting Thermohm

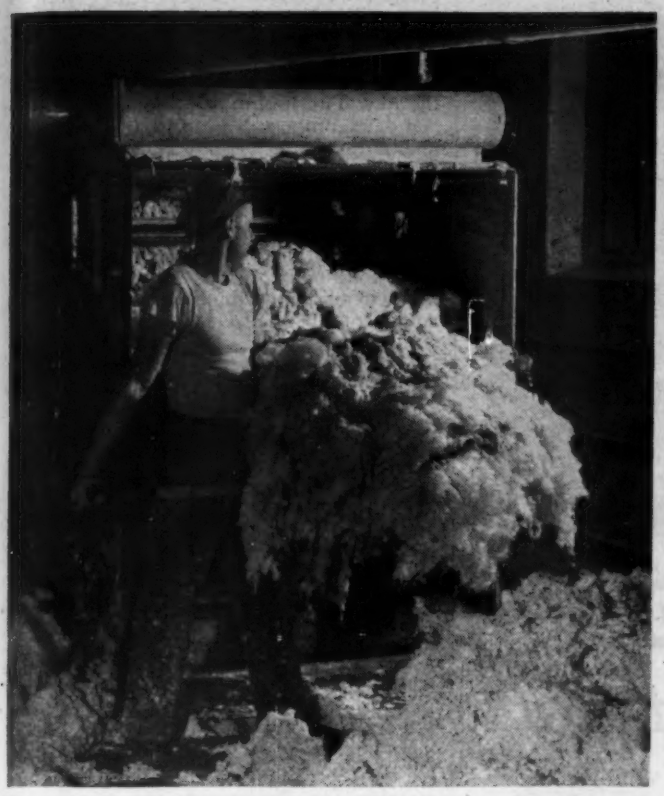
Leeds & Northrup Company, 4934 Stenton Avenue, Philadelphia 44, has introduced a new type of industrial Thermohm (resistance thermometer bulb) which extends the range of L&N resistance thermometer recorders and controllers to temperatures as high as 1000° F.

This Thermohm is amply accurate for the more exacting industrial requirements. Speed of response is much faster than that of usual heavy-gauge couples and is matched only by small pipe-type or fine-wire couples. Rugged, durably constructed, this Thermohm is easy to install in ovens, tanks, ducts or pipe lines.

Its own corrosion-resisting stainless tube gives ample protection for normal pressures and non-corrosive fluids. Like any thermocouple or thermometer bulb, the Thermohm can be used with protecting wells where required. The tempera-



Leeds & Northrup Thermohm



From Raw Material to Finished Product

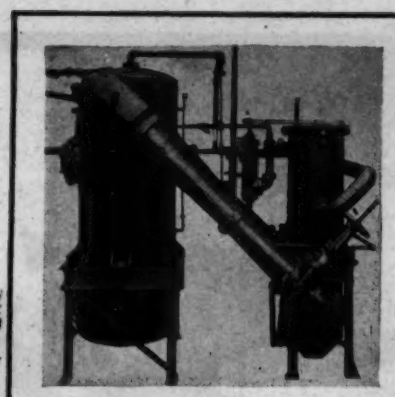
ALL GARLOCK packings are *quality controlled* from raw material to the finished product. Photo shows the first step in the preparation of crushed asbestos fibre for use in the manufacture of Garlock asbestos packings. One of these Garlock products is GARLOCK 234 which is recommended for rotary or centrifugal pumps handling hot water, cold water, caustic solutions or weak acids. For long, dependable service specify GARLOCK 234.

THE GARLOCK PACKING COMPANY
PALMYRA, N. Y.
Tulsa, Okla. Houston, Texas
Los Angeles, Calif.

60th Anniversary 1887—1947

GARLOCK 234 Braided Asbestos Packing in Coil form. GARLOCK 244—Coil with wire. GARLOCK 239—Rings. GARLOCK 249—Rings with wire.

GARLOCK 234



ORDER YOUR CHILLVACTOR NOW

For Chilled Water Next Summer

Every year when the warmest weather comes in July and August, we have several frantic appeals from production men in petroleum and process industries for CHILL-VACTOR equipment to maintain proper temperatures in certain solutions, in chilled water for air conditioning, for chilling various reaction vessels, and for special cooling requirements.

The Croll-Reynolds CHILL-VACTOR is one of the most dependable and efficient units available for cooling requirements in the range above 32° F. While there are a great many outstanding advantages, there are a few limitations. In recent years the most serious one seems to be the fact that these units are not made in quantity and available for immediate shipment. While they are mostly made from standard parts, each unit is custom built to a certain extent in order to provide maximum dependability and efficiency under individual operating conditions.

Under present manufacturing schedules, it requires approximately three months to design, build and ship a complete CHILL-VACTOR unit. This means that **immediate** action is advisable for those who want to be ready with new cooling capacity for the next warm weather season.

The Croll-Reynolds CHILL-VACTOR has no moving parts other than a standard chilled water circulating pump. Water is the only refrigerant. Maintenance and repair costs are therefore practically nil. Operating costs are usually less than for other types of refrigerating equipment where ample condenser water is available. Inquiries on this type of equipment will receive the careful attention of engineers with many years specialized experience in this field.

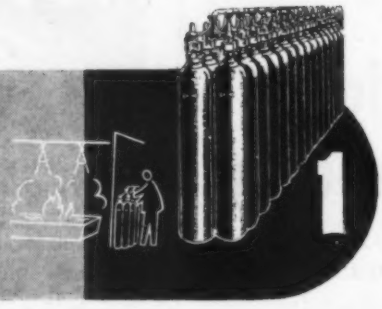
CROLL-REYNOLDS

CROLL-REYNOLDS CO.

17 JOHN STREET, NEW YORK 7, N. Y.

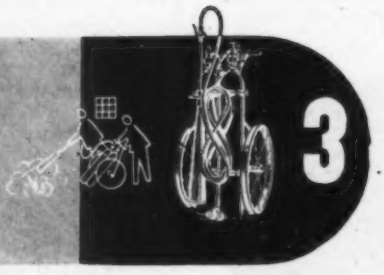
CHILL VACTORS STEAM JET EVACTORS CONDENSING EQUIPMENT

FIRE BRIGADE...
...modern style



INSTANT ACTION against fire with the original C-O-TWO pressure-operated type, built-in system. Engineered to protect one or several spaces, electrical equipment, paint, oil, lacquer, alcohol and other highly flammable liquids and stored materials. Operated in combination with smoke detector and thermostat controls.

SIMILAR HAZARDS constantly vulnerable are adequately protected by C-O-TWO hose reel units ready for instant action with a fast uncoiling hose ready to blast fires with dry, sub-zero carbon dioxide gas, the fastest non-damaging, non-conducting, non-deteriorating fire extinguishing agent.



HURRY UP CALLS are covered by C-O-TWO wheeled units. Sturdily made of durable materials and easy to operate, fast and safe in single or double cylinder sizes equipped with adequate length of hose. Rolled easily and quickly by one man to scene of fire and take minimum space when not in use.

C-O-TWO FIRE EQUIPMENT COMPANY

NEWARK 1 • **NEW JERSEY**

Sales and Service in the Principal Cities of United States and Canada

AFFILIATED WITH PYRENE MANUFACTURING COMPANY

ture coil is thoroughly protected against contamination by moisture or gases.

Like L&N thermocouples and lower-temperature resistance thermometers, the new Thermohm is equipped with the standard universal head that facilitates installation and connection. Standard lengths are 6 and 12 inches.

CHECK THE CARD AT PAGE 220

11—Rotary Pumps

Worthington Pump and Machinery Corporation, Harrison, New Jersey, has introduced a new line of general-service rotary pumps, the GA Rotary type. Six sizes are available in various mountings for capacities up to 51 gallons per minute and 100 pounds per square inch.

These small-capacity rotary pumps have been designed for durability under severest service requirements, and adaptability to variety of such requirements offered by industries. Design features include four-bearing construction and special herringbone rotors. Built-in pressure lubrication eliminates all external lubrication and the danger of liquid contamination. A production line item, the GA Rotary in its simplicity of design also offers easy maintenance and low first cost.

Models are available footmounted or flange mounted with adjustable stuffing box and mechanical seal. They are also available with or without oversize unloader type relief valve. All models are suitable for direct coupling to electric motors of any make.

CHECK THE CARD AT PAGE 220

12—Liquid-Level Transmitter

Taylor Instrument Companies, Rochester 1, New York, has announced the Taylor 229RF Liquid-Level Transmitter, a single compact unit which measures the level of the liquid and transmits an air pressure proportional to this level to the remotely located instrument.

This transmitter is described as particularly applicable for measuring the level of viscous liquids or liquids with solids in suspension; the level measurement of liquids or solutions which solidify in the usual pressure or manometer connections; and relatively low pressure measurement (up to 17 psi) of corrosive gases or liquids for which the diaphragm type element is particularly suited.

The single unit consists of a metal diaphragm in a 3-inch 150 psi stainless-steel flange which can be attached to the bottom or the side of an open or vented tank.

This transmitter operates on the force-balance principle. Being a 1 to 1 transmitter, the output air pressure is equal to the applied pressure or liquid head and has a range of 450 inches of water with an accuracy of plus or minus 1 inch of water.

Bulletin 98069 illustrates and gives complete and detailed information regarding this new transmitter.

CHECK THE CARD AT PAGE 220

13—Pump Stroke Adjustment

Milton Roy Company, 1383 East Mermaid Avenue, Philadelphia 18, has issued a new Catalog No. 146, which describes an improved type stroke adjustment for regulating, while running, the volume of liquid pumped by standard Milton Roy chemical pumps. This micro-indicating adjustment, supplied as original equipment, provides precise control of plunger stroke by means of a hand wheel. A vernier dial permits quick and convenient resetting to any pre-determined

Bigger Filters for BIGGER M·E·K PLANTS

THE advantages of fewer filter units installed in big M·E·K Lube Plants are:—

Lower installed cost
More time on stream
Lower operating costs
Less maintenance

The newest General American Conkey solvent dewaxing filter now being constructed is a BIG filter, General American's biggest to date – providing 625 square feet of filter surface in rotary drum 10′ diameter by 20′ long.

This large size is practicable due to the Conkey structurally ribbed rotary drum construction which adds size option to the many other advantages of its design.

Bulletin #104, now in preparation, will outline other advantages of this largest size filter unit. Write for your copy which will be mailed when available.

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TRANSPORTATION CORPORATION
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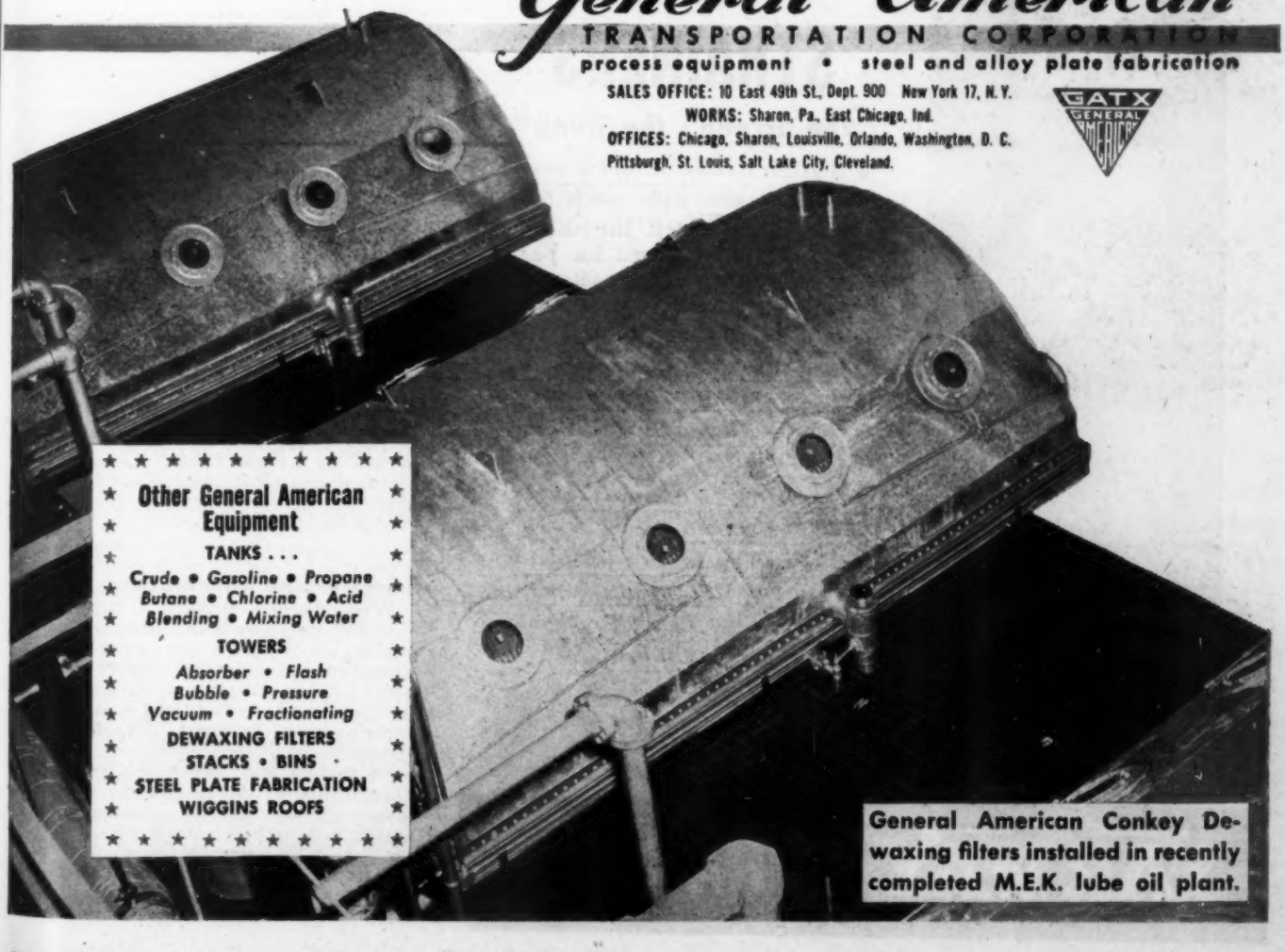
GATX GENERAL AMERICAN



Other General American Equipment

TANKS . . .
Crude • Gasoline • Propane
Butane • Chlorine • Acid
Blending • Mixing Water

TOWERS
Absorber • Flash
Bubble • Pressure
Vacuum • Fractionating

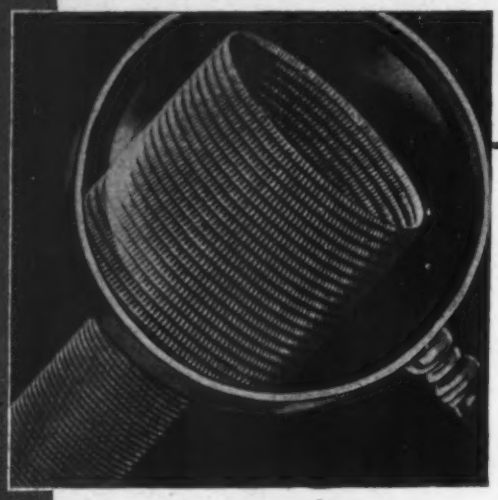
DEWAXING FILTERS
STACKS • BINS
STEEL PLATE FABRICATION
WIGGINS ROOFS

General American Conkey Dewaxing filters installed in recently completed M.E.K. lube oil plant.

PREVENT

Rust·Scale·Dirt

FROM CLOGGING
STEAM EQUIPMENT



YARWAY

STRAINERS

"The Screen's the Thing"

- *A High Grade Monel Woven Wire Basket* with mesh fine enough to catch the dirt—yet ample free area for passage of clean condensate, oil and other fluids.
- *Body Cadmium plated for protection against corrosion and for better appearance.*
- *Readily Removed Steel Blow-Off Bushing.*
- *Bushing Automatically Aligns Screen.*
- *Thousands in Use—Sold by Over 100 Mill Supply Houses.*
- *6 Sizes ½" to 2" for Pressures up to 600 lbs.*

See Your Supply House or write for Bulletin S-201

YARNALL-WARING COMPANY

128 MERMAID AVENUE PHILADELPHIA 18, PA.

YARWAY STRAINERS

stroke length, to within 1/750 of the length of the stroke without stopping the pump. Models also are available with automatic motorized stroke adjustment. These pumps are recommended by the manufacturer especially for controlled-volume pumping where it is desired to pump one or more liquids in precisely metered quantities, and for pumping in volumes as small as one pint per hour. Valves and parts are supplied in any metal or alloy that can be cast and machined, and in certain plastic materials, to meet practically all corrosion-resistance requirements.

CHECK THE CARD AT PAGE 220

14—Fuel Improver

Enjay Company, 26 Broadway, New York 4, is distributing its Technical Bulletin 101, "Paradyne Fuel Improver —Its Effect on Gasoline Performance." It describes a new product whose principal purpose is to improve performance of motor gasoline, functioning as a gum solvent and "upper lubricant." The material inhibits formation of gummy deposits, aids in removing deposits already formed around valves, and helps to improve lubrication in the upper area of an engine, it is set out in the bulletin. The product is to be marketed to refiners and compounders to be incorporated in gasoline, or to be packaged and sold for adding to gasoline at the pump.

CHECK THE CARD AT PAGE 220

15—Instruments

The Foxboro Company, Foxboro, Massachusetts, has available Bulletin 397, which illustrates and describes its Dynalog Electronic Instruments, including important features recently added.

CHECK THE CARD AT PAGE 220

16—Flowrator Dimensions

Fischer & Porter Company, Hatboro, Pennsylvania, has issued a bulletin containing complete dimensions covering every available combination of its Series 700 Flowrator (rotameter) instrument. Available for limited distribution to engineers and piping-layout draftsmen, it is presented as an effort to reduce to the handiest possible form the necessary dimensional information.

CHECK THE CARD AT PAGE 220

17—Agriculture

Shell Oil Company has presented the information concerning its new agricultural laboratory and experimental farm at Modesto, California, in a book, "Better Farming Through Research." An outline of the activities of the venture as well as a presentation of the staff is given along with illustrations to reveal applications of chemicals in the several contributions to agriculture.

CHECK THE CARD AT PAGE 220

18—Turbine Maintenance

Westinghouse Electric Corporation, Box 868, Pittsburgh 30, has issued a new booklet (B-3747) containing practical information necessary for proper maintenance of general-purpose turbines. The booklet first describes the steam turbine, explains how it operates, and tells how it should be installed. Directions for piping, joint sealing and lubricating are given. Dismantling of the turbine for complete inspection once a year is recommended, and points to be checked and recorded are listed. Necessary repairs and adjustments are discussed under the heading of the various parts—rotor, bearings, glands, governors, nozzles, overspeed

Which tubing will last the *Longest?*

Photo: Courtesy Henry Vogt Machine Co., Louisville, Ky.

The corrosive liquids and vapors passing through heat exchangers, condensers and evaporators in petroleum refineries and chemical plants are a constant threat to continuous operation because of the possibility of premature tube failure. Aside from interruption in service, leaky tubing may involve product contamination, injury to personnel and equipment, and, of course, the expense of replacement.

Bridgeport considers the question of service life so important that a consistent program of corrosion research is maintained, involving extensive laboratory work as well as actual field and service tests to help customers obtain longer tubing life.

Although Bridgeport's tubing is used in thousands of plants — i.e., Duplex Tubing for ammonia refrigeration; Arsenical Admiralty in oil refineries; Arsenical Muntz* for severe sulfur corrosion; Cuzinal (aluminum brass) for resisting air impingement corrosion; Duronze IV** (aluminum bronze) for hot brine and seaboard power plants; Cupro Nickel for high water velocities, etc. — each corrosion problem is considered individually. Bridgeport's Technical Service Department works closely with customers in solving corrosion problems and trying out test lots of tubing and corrosion test specifications under actual operating conditions.

Contact the nearest Bridgeport office for this service, and write for free copies of Duplex Tubing Bulletin No. 746 and the 112-page Condenser Tube Manual.

*Reg. U. S. Pat. No. 2118688 **Reg. U. S. Pat. No. 2083390

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To Improve Cast . . .

Fluorescent Green H.W. 185% produces a "Pennsylvania Cast" in lube oils. It is an additive which definitely will improve the salability of your lubricants.

Fluorescent Green: H.W. 185%

Solubility ---------- readily and complete
Color ---------- unchanged by transmitted light

STABILITY:

light ---------- fast
heat ---------- stable
storage ---------- stable

TESTS:

carbon residue ---------- unchanged
fire ---------- unchanged
flash ---------- unchanged

Leading lube refiners in the United States and abroad have used Wilmot & Cassidy products for fifteen years. *For samples, prices, and information please write to:*

Wilmot & Cassidy, Inc.

108 Provost St. Brooklyn 22, New York

trip, speed changer, reduction gears, and oil parts. The final chapter gives emergency hints for quick reference if the turbine is not operating properly.

CHECK THE CARD AT PAGE 220

19—Instruments

Wheelco Instruments Company, 847 West Harrison Street, Chicago 7, has available a 20-page educational bulletin, No. 5, on automatic temperature-control systems. Charts, tables and diagrams explain measurement and automatic control, and selection of control systems for process applications. Thermocouples and tubes are discussed separately.

CHECK THE CARD AT PAGE 220

20—Water Temperature Control

The Powers Regulator Company, 2720 Greenview Avenue, Chicago 14, has issued a new 16-page catalog, No. 3035, on regulators and thermostatic water-mixing valves for water-temperature control.

CHECK THE CARD AT PAGE 220

21—Eye Protection

Mine Safety Appliances Company, Pittsburgh 8, has issued a new catalog illustrating and describing its line of eye-protective devices.

CHECK THE CARD AT PAGE 220

22—Hose Couplings

Hose Accessories Company, Lehigh at 17th, Philadelphia 32, has available a 20-page booklet on use of hose couplings and fittings. It presents hints and information on care of hose.

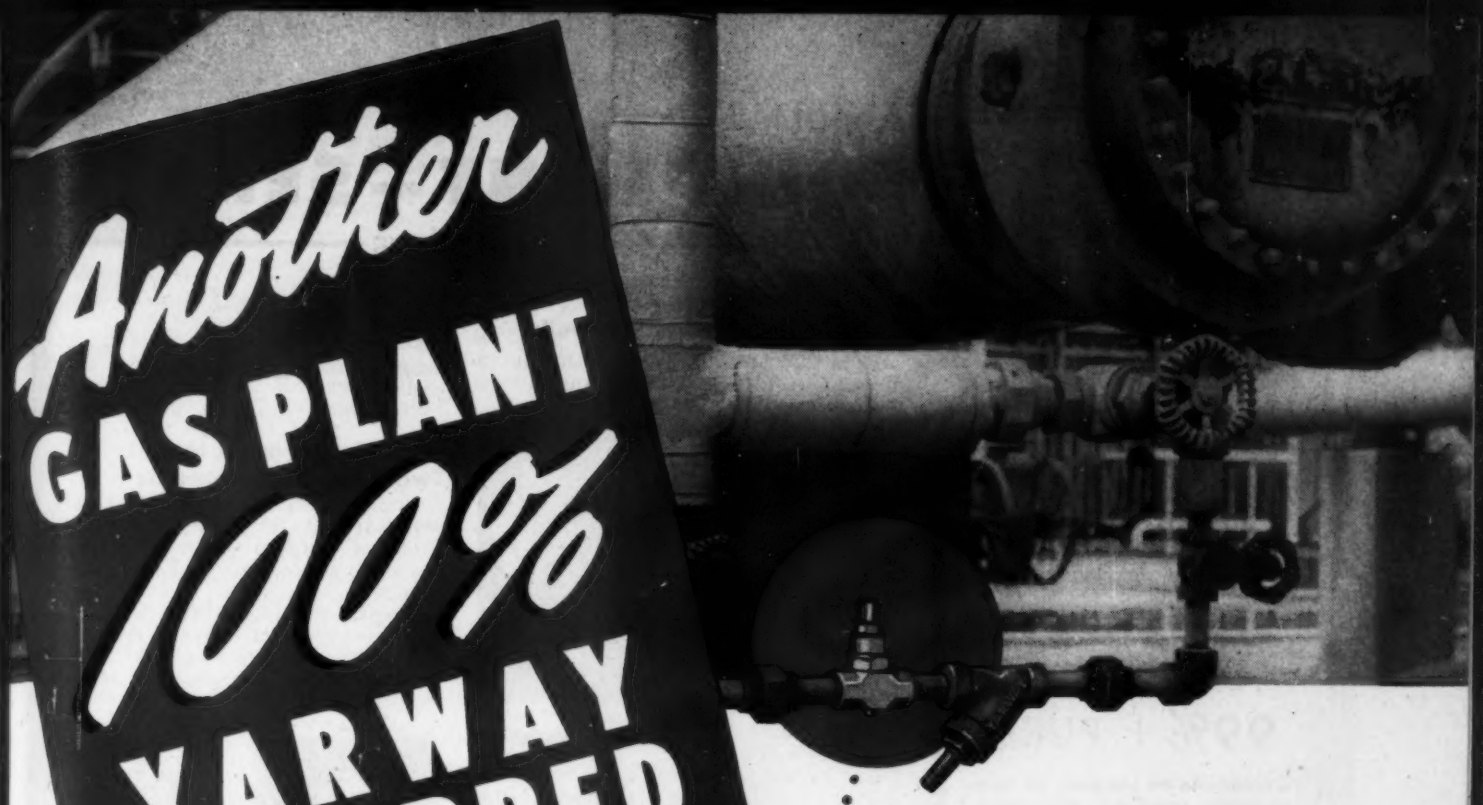
CHECK THE CARD AT PAGE 220

23—Organic Nitrogen Compounds

Carbide and Carbon Chemicals Corporation, 30 East 42nd Street, New York 17, has issued a new 31-page booklet, Form 4770, "Organic Nitrogen Compounds," which presents in detail the properties, specifications, and uses of the alkyl amines, alkylene amines, alkanolamines, and acetoacetarylamides. In chart form it gives their physical constants, comparative hygroscopicities, and neutralization curves. These compounds are used by various industries for many purposes such as emulsifying, neutralizing, synthesizing, dissolving, and saponifying.

The organic nitrogen compounds are extremely reactive; with acids they form salts or soaps; with esters, anhydrides, and acyl halides they form substituted amides; and with ketones, aldehydes, and alkyl halides they form other important derivatives. Many of the present applications of amines arise from their ability to neutralize acidic materials, such as in aqueous systems as corrosion inhibitors; in purification of gases by the removal or concentration of hydrogen sulfide or carbon dioxide; and in the formation of emulsifiers for polishes, cosmetics, insecticides, water paints, and cutting oils. They are widely used as intermediates in the production of dyestuffs, detergents, textile softeners, cement setting compounds, photographic and rubber processing compounds, antimalarials, local anesthetics, and pharmaceuticals. Newer uses for amines include ion-exchange and water-softening resins, germicides, flotation agents, petroleum emulsion breakers, gasoline additives, and catalysts.

CHECK THE CARD AT PAGE 220



Another GAS PLANT 100% YARWAY EQUIPPED

COLUMBIA GAS TREATING PLANT OF ARKANSAS FUEL OIL CO. USES ALL YARWAY IMPULSE STEAM TRAPS

Again and again you hear it—Yarway Impulse Steam Traps used throughout the plant!

Here at this large southwestern plant nearly 100 Yarways are on the job on re-boilers, separators, steam turbine-driven pumps, reciprocating steam pumps and elsewhere.

Yarways are popular for these reasons:

First—they're good for *all pressures* within a wide range without change of valve or seat.

Second—Yarways speed up heating and keep equipment hotter because they discharge *continuously* on heavy condensate loads, at short intervals in lighter loads . . . keeping lines free of condensate.

Third—simple design, only one moving part, makes maintenance easy.

Fourth—it is frequently cheaper to install a new Yarway than to repair an old type trap.

See your Supply House, or write for Yarway Bulletin T-1739.

YARNALL-WARING COMPANY
128 Mermaid Avenue, Philadelphia 18, Pa.

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YARWAY IMPULSE STEAM TRAP

ESPECIALLY RECOMMENDED FOR USE IN THE

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COPPER SULPHATE

is preferred by leading refineries because of its high copper content, dependable uniformity and exceptional purity and freedom from foreign and inert matter.

99%+ PURE

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All Meshes
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WIRE CLOTH COMPANY, INC.
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About EQUIPMENT—SERVICE Suppliers

L. M. Morley Elected Vice President of Honeywell

L. M. Morley, vice president in charge of sales for The Brown Instrument Company, Philadelphia, has been elected a vice president of Minneapolis - Honeywell Regulator Company. The Brown company is a wholly-owned subsidiary of the latter company, and Morley will continue his position and activities at Philadelphia.

L. M. Morley

A graduate of the University of Pennsylvania, Morley joined The Brown Instrument Company in 1919 as a salesman. He was made sales manager in 1934, and became a vice president in 1935 when the two mentioned companies were merged.

Rockwell Manufacturing Company Announces Two Appointments

C. A. Wiken, for the past eight years chief engineer of Delta Manufacturing Division of Rockwell Manufacturing Company in Milwaukee, has been promoted to vice president in charge of engineering for Rockwell Manufacturing Company.

A. E. McIntyre, who for the past several years has been manager of the Nordstrom Valve Division plant, Oakland, California, has been transferred to Pittsburgh as general manager of the Pittsburgh Equitable Meter Division. McIntyre came to the Nordstrom Valve Division after 17 years with Caterpillar Tractor Company, where he was factory manager. He will continue to serve as manager of the Nordstrom Valve Division.

American Meter Company Announces Two Promotions

Appointment of William G. Hamilton, Jr., as sales manager, and of Arthur F. Benson as production manager, has been announced by American Meter Company.

Hamilton's connection with the company dates from 1927. He became assistant manager of the Philadelphia factory in 1941, and since September, 1945, has been manager of Philadelphia operations.

Benson has been with American Meter Company since 1930, and under his new assignment will continue as chief engineer, a position he has filled since 1943.

A. F. Benson

W. G. Hamilton, Jr.

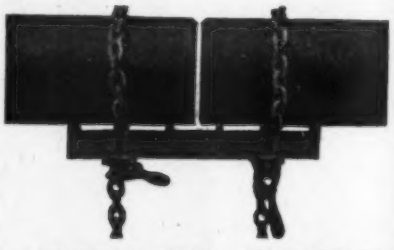
Ethyl Corporation Makes Two Sales Appointments

Appointment of Oscar B. Lewis as assistant sales manager, and E. Oliver Jones as manager of customer services has been announced by Ethyl Corporation.

Lewis, who has been manager of safety for the past 12 years, joined Ethyl Corporation 20 years ago. After special assignments in England and service as manager of the Baltimore sales division

E. O. Jones

O. B. Lewis



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Faster... Easier... Truer
WITH
JEWEL PIPE CLAMPS

They align pipe perfectly and hold it secure for tacking. On in a minute—off in ten seconds. Two sizes, ½ to 8 in. — 8 to 16 in. Rigid construction resists strain, heat, and abuse.

Other Jewel Clamps for Ells, long and short turn — for Flanges — for Headers. Also Pipe Marker, eliminates patterns for holes and saddles.

So simple — so easy to use — Jewel Tools are proving big time-savers in refinery piping fabrication and erection.

Write Today for Details and Prices

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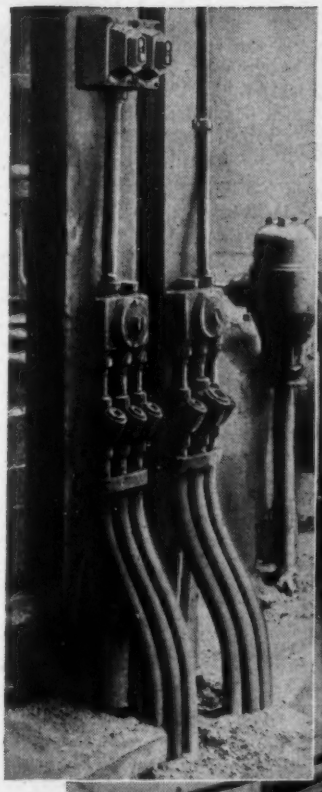
Where Rustable Conduit Fails in months

Everdur lasts for years...



This concentration of Everdur Electrical Conduit and Everdur fittings at left is located in a moist alcove between cooling towers, yet shows no evidence of corrosive attack.

Everdur Electrical Conduit installation at the Torrance Refinery of General Petroleum Corporation after 8 years service in corrosive atmosphere.

A GOOD EXAMPLE of the ability of Everdur* Copper-Silicon Alloy to withstand severe corrosion is this conduit installation of the Torrance Refinery of General Petroleum Corporation.

Because corrosion-resistant materials were not available, several installations of ordinary conduit were made during the past five years. In the vicinity of the cooling towers, the action of spray from chemically treated water is so severely corrosive that one such installation was replaced twice in two years.

How does Everdur Electrical Conduit stand up under such conditions? This durable material installed eight years ago shows *no effects other than discoloration.* Accordingly, General Petroleum now has definite plans for replacing corroded conduit with Everdur. 46194

*Reg. U. S. Pat. Off.

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for 5 years, he transferred to New York in 1935 to organize the company's safety program with customer companies. Jones, whose new duties will include direction of safety programs, joined the company in 1932, and served as a safety engineer until he became a member of the staff of PAW at Washington. Since that war service he has been a special assistant to Julian J. Frey, general sales manager.

Marshall Sales Manager Asbestos Products Division

George W. Marshall, Jr., has been named general sales manager of the Asbestos Products Division of Raybestos-Manhattan, Inc. He will continue as general manager of the asbestos textile and packing division, and will direct sales activities of the equipment sales division. He will be located at 445 Lake Shore Drive, Chicago.

Earl H. Bodinson Joins Hammond Iron Works

Earl H. Bodinson has joined the sales staff of Hammond Iron Works of Warren, Pennsylvania, and Port Neches, Texas. He will make his headquarters in the company's New York offices, 630 Fifth Avenue, where he will assist R. E. Kramer, vice president in charge of sales.

E. H. Bodinson

Bodinson, a graduate of University of Illinois, has been identified with the metallurgical and sales side of the steel industry since 1928, with Illinois Steel Company, Jos. T. Ryerson Sons, and lately with Pittsburgh Steel Company.

Roensch Joins Ethyl Detroit Engineering Staff

Max M. Roensch has joined Ethyl Corporation as a member of the engineering department of the research laboratories in Detroit. As one of the three coordinators on the staff of J. B. Macaulay, director of engineering research, Roensch will be responsible for research and development pertaining to the power, economy and durability of engines. In the field of internal-combustion-engine research for many years, he is widely known in both the automotive and petroleum industries through his former association with Chrysler Corporation Engineering Laboratories and his more recent position as chief engineer of Cleveland Graphite Bronze Company. He is a member of the Engineering Society of Detroit and the Society of Automotive Engineers in which he has served on many technical committees. After graduating from The Rice Institute he received his M.S. degree from the University of Michigan in 1926.

M. M. Roensch

Royse Representing Fluor Corporation at Houston

C. F. Royse has been appointed district engineer for The Fluor Corporation, in charge of the Houston office.

Royse for the past five years was with Griscom-Russell Company in the Houston area, and before that for five years was with Humble Oil & Refining Company. He previously had graduated from The Rice Institute with a degree in chemical engineering.

C. F. Royse

California Company Makes Finned Tubing

Cal-Fin Company, 1128 Mission Street, South Pasadena, California, is manufacturing a finned tubing under license from the extended surface division of David E. Kennedy, Inc. Applications in the refining industry are for furnace tubes in convection banks and for fan-cooled units for removing heat from jacket water, hot gases and in condensing steam.

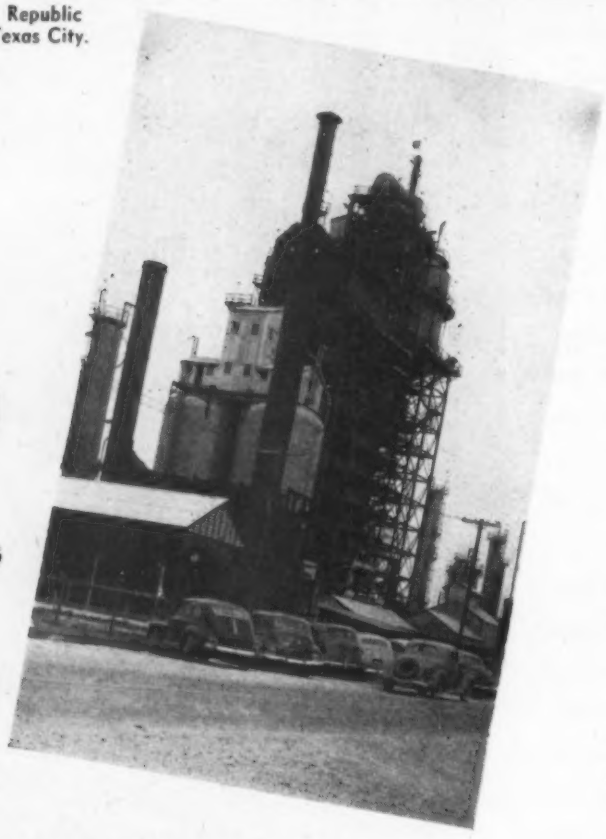
Alloy Steel Products Company Adds Two to Sales Force

Herbert V. Evans and H. C. Grieme recently joined the sales department of Alloy Steel Products Company, Linden, New Jersey.

Evans was with Lebanon Steel Foundry prior to service in the Army Air Forces. Recently he represented Anti-corrosive Metal Products Company in New England.

Grieme, recently with the Bureau of

Cat Cracker Republic Refining Co., Texas City.



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TUBE ALIGNING-ANCHOR CLAMP MAKES *Unibolt* STREAMLINED RETURN BENDS *still more desirable*

1

The Tube Aligning-Anchor Clamp fits around the upset tube ends to provide a rigid support for the tubes and serves as an anchor ring for the return bend holding member. The return bend body may be easily removed and just as easily re-installed because the tubes are thus held in a fixed position.

2

The Tube Aligning-Anchor Clamp is held in position around the upset tube ends by a single bolt. The return bend body may be removed without disturbing the clamp . . . it remains in position until the tubes require replacement. Note that the clamp fits snugly around the tube ends to prevent their moving out of position when the bend is removed.

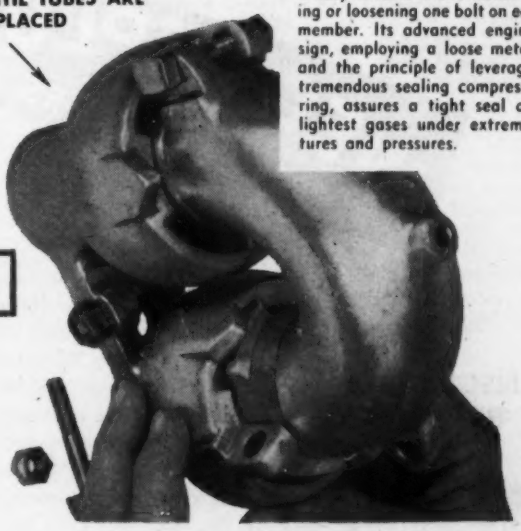
It is unnecessary to remove the return bend holding members when the bend body is removed although it can be easily accomplished at will since the holding member is made to slip on and off the anchor clamp. This photograph shows one of the holding members being stripped off.

3

CLAMP IS NEVER REMOVED UNTIL TUBES ARE REPLACED

The UNIBOLT Streamlined Return Bend is easy to remove and install by tightening or loosening one bolt on each holding member. Its advanced engineering design, employing a loose metal seal ring and the principle of leverage to apply tremendous sealing compression on the ring, assures a tight seal of even the lightest gases under extreme temperatures and pressures.

4



THORNHILL-CRAVER CO., HOUSTON, TEXAS

UNIBOLT
REFINERY FITTINGS

Ships, United States Navy, before that was engaged in purchasing work with Chemical Construction Corporation, H. K. Ferguson Company, and Campbell Soup Company.

Paxson Representing Taylor Instrument at St. Louis

Robert E. Paxson recently has been assigned to represent Taylor Instrument Companies in the Midwest, with headquarters at St. Louis.

Paxson graduated with a degree in chemical engineering from University of Colorado, and was chief chemist for a sugar company in Wyoming before serving in the war as a Navy ordnance officer. Since discharge he completed an 11-month industrial training course at Taylor Instrument Companies' home office in Rochester, New York.

Republic Supply Company Opens Denver Sales Office

Republic Supply Company, Houston, announces extension of its supply service to the upper Mountain States area with the opening of a sales office at Denver. The new office is located at 639 Continental Building and Denver H. Hyler is in charge.

D. H. Hyler

Hyler has been transferred from the company's district office in Wichita Falls where he handled refinery and industrial sales. Prior to joining Republic Supply Company in 1945, Hyler spent 30 years in active refinery operations and was connected with Continental Oil Company for 21 years, and for 19 years he was in charge of that company's Wichita Falls refinery.

With the opening of the new Colorado sales office, Republic Supply Company now serves the oil industry with more than 50 sales and service points in ten states including: Arkansas, Colorado, Illinois, Indiana, Kansas, Louisiana, Mississippi, New Mexico, Oklahoma and Texas. General offices are maintained at Houston.

Schneider Joins Pennsylvania Salt Chemical Sales Force

John H. Schneider has joined the sales service staff of the special chemicals division of Pennsylvania Salt Manufacturing Company.

Schneider, a graduate of University of Minnesota, in aeronautical engineering, served as a maintenance and repair officer with the air forces in the European theater, and later was assigned to an American Army team to investigate aircraft developments in Germany. Before his release from service, as major, he spent a year working on development of jet aircraft at Wright Field, Dayton, Ohio.

Crouse-Hinds Company In Business 50 Years

In recognition of its half century as a business institution, Crouse-Hinds Company, Syracuse, New York, made distribution of a book "Fifty Years," in January, 1947. The company is the result of a partnership between Huntington B. Crouse and Jesse L. Hinds, whose venture was housed in space that involved a rent of $33.33 per month, including heat and power. The history of growth of this partnership into a corporation includes many brief autographical references and ends with a listing of the full roll of employes as of December 14, 1946.

Jones & Laughlin Opens New Oklahoma Store

Jones & Laughlin Supply Company has opened a new store at Pauls Valley, Oklahoma. William D. Breedlove, formerly with the company's Seminole, Oklahoma, store, is manager of the new store, operating as part of the Central and Southern Oklahoma district, under supervision of L. R. Roberts, district manager at Wewoka.

R. A. Weber recently has been named field salesman for the company in the Hutchinson, Kansas, area.

Allen Appointed General Sales Manager for Kerotest

George R. Allen has been appointed general sales manager for Kerotest Manufacturing Company, Pittsburgh. He has been with the company several years; recently as manager of the brass valve division. He is a graduate of University of Michigan.

George R. Allen

The appointment, it was announced, was made in a move to combine sales operations of the company for both brass and steel valves under one head. Grow-

The All-Purpose Valve

Has Large Port Openings

WEDGEPLUG ALL-PURPOSE STEEL VALVES

NEGLIGIBLE PRESSURE DROP

Large port openings are provided, thereby assuring little pressure loss through a WEDGEPLUG Valve. Openings in percent of pipe area are:

Series 15—56% to 96%
Series 30—81% to 96%
Series 60 and up—100%

WEDGEPLUG Valves are so designed that pressure-drop is reduced to a minimum, and is definitely not a limitation.

WEDGEPLUG offers the only real "All-Purpose" Valves on the market today—through the development of a simple, patented Dry Plug Valve that combines the best features of a Plug Valve with those of a Gate Valve: Large port openings; wedging action; raised seats; and, an easy-to-operate mechanism that *lifts, turns, and reseats the Plug in one operation.* A Plug-type Valve that can take the place of a Gate Valve. Tried and proved under widely-varying service conditions. Write for Bulletin No. 545.

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OKLAHOMA CITY—WICHITA—TULSA—ODESSA—AMARILLO—CHARLOTTE
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NH VALVES

for the oil industry cover a wide range of specialized productions. Gate, Stop and Check Valves for high pressures and temperatures, steam oil, gas and for general purposes. Also Newman-Milliken Glandless Lubricated Plug Valves for all oilfeld and refinery services. Working pressures to 5,000 lbs. per square inch (tested to 10,000 lbs. per square inch Kerosene). Made in various metals to suit particular services.

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Physical Constants
OF
HYDROCARBONS

By GUSTAV EGLOFF, Director of Research, Universal Oil Products Company, Chicago.
A. C. S. Monograph No. 78

- **VOLUME I—Paraffins, Olefins, Acetylenes, and Other Aliphatic Hydrocarbons.** *403 pages, $10.00*
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- **VOLUME III — Mononuclear Aromatic Compounds.** *672 pages, $16.00*
- **VOLUME IV—Polynuclear Aromatic Compounds**

Organic research chemists, development engineers, and physical chemists in the many industries in which aromatic compounds are involved will find this volume of critically evaluated data a noteworthy and highly useful addition to the literature. It will be indispensable to workers in the petroleum and related fields and also to those in the coal-tar, dye, pharmaceutical and synthetic chemical industries. This monumental series, of which this is the fourth volume, will also be of prime interest to technical librarians, professors of chemistry, and advanced students specializing in organic synthesis.

The organization of Volume IV is the same as that of the three preceding volumes. The name and structural formula, together with melting point, boiling point, density and refractive index values as determined by numerous investigators at widely different atmospheric pressures, are given for all known compounds whose chemical nucleus consists of more than one ring. Ideal values as selected by the author are indicated for each compound on which a large number of determinations is reported. The material is copiously documented; additional data are presented when available.

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Model 33-S-9 in mild steel. Steam jacketed. Cap. 2,600 G.P.H.

Sizes from 1 sq. ft. to 150 sq. ft. of filtering area.

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Cake will not break or slip during wash-through with solvents or blow-down with gas or air pressure.

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RUST-OLEUM
POSITIVELY PREVENTS RUST

PROTECTS YOUR METAL SURFACES FROM CORROSION

(See Page 968—Refinery Catalog)
RUST-OLEUM CORPORATION
EVANSTON, ILLINOIS
TEXAS BRANCH
1935 Commerce St., Dallas, Texas

ing emphasis on LP gas also was a factor which helped to set a company policy of over-all sales operations, it was said, together with a noted growing trend of intermingling brass and steel in smaller valve applications in many industries.

Black, Sivalls & Bryson Announces Sales Changes

Black, Sivalls & Bryson has announced three changes in sales personnel, made necessary by the recent death of J. A. Grubb, who had been regional manager for the company in Kansas and the Northwest.

Ed Nelson

Ed Nelson has been transferred from Kansas City to Great Bend, Kansas, where he will direct Kansas operations for the company.

Marion Miller, who had been Grubb's assistant in the Wichita sales office, now is in charge of that office.

Lee Kelton has been made district manager in charge of the Connor Tank Division in its operations in Wyoming and Colorado.

International Nickel Company Names Two Vice Presidents

H. J. Fraser and H. J. French have been elected as additional vice presidents of The International Nickel Company, Inc. Fraser has been assistant vice president since June, 1943. Both he and French are assistant vice presidents of The International Nickel Company of Canada, Limited, the parent company.

Fraser joined the Huntington Works of The International Nickel Company in May, 1923, following his graduation from Queen's University, Kingston, Canada. After serving for some years in various technical and operating capacities, he was promoted to the New York office in February, 1935, becoming assistant manager of production department. He has been assistant vice president of both companies since June, 1943.

French, after receiving the degree of metallurgical engineer from the School of Mines, Columbia University, in 1915, served with the U. S. Army during World War I. In 1919 he joined the metallurgical staff of the U. S. Bureau of Standards in Washington, subsequently becoming assistant chief of the Division of Metallurgy. He became associated with International Nickel in 1929 and was for years in charge of alloy-steel development. Assistant manager of the development and research division since September, 1943, he recently was appointed an assistant vice president of The International Nickel Company of Canada, Limited.

Pittsburgh Steel Company Revives Marketing Subsidiary

Pittsburgh Steel Products Company, subsidiary of Pitsburgh Steel Company, has been revived and expanded to become the marketing division for the parent company's products.

Top management of the subsidiary is the same as for the parent organization, headed by Joseph H. Carter, president; J. K. Beeson, executive vice president; Joseph A. Voelker, vice president; and J. H. Morrison, vice president in charge of operations.

T. C. Phillips

R. L. Glose

Thomas C. Phillips and Robert L. Glose have been elected vice president in charge of sales and general manager of sales, respectively, of the subsidiary organization.

WANTED—MECHANICAL OR METALLURGICAL ENGINEER—familiar with the inspection of refinery fractionating equipment, furnaces, heat exchangers and pressure vessels and having experience to qualify for making recommendations for renewal or repairs to the equipment. Experience on catalytic cracking units preferred, but not essential. In replying, please send full details of experience and education. Snapshot (if available). Address: Box 10-R, c/o Petroleum Refiner, Houston, Texas.

WANTED

Graduate chemical engineer with experience in petroleum refining for teaching fundamental principles of chemical engineering. Position permanent in well established engineering school in West. Give age and experience in first letter. Address: Box 3-R c/o Petroleum Refiner, Houston, Texas.

ENGINEER

Engineering firm, headquarters New York City, requires graduate engineer experienced construction and mechanical design petroleum refineries or power plants. Fluent French and English. Two years foreign assignment in large European city. Address: Box 2-R, c/o Petroleum Refiner, Houston, Texas.

FOR SALE—300 H.P. Clark RA3 Gas Engine Driven Compressor with or without compressor cylinders. Brand new. R. H. Armstrong, Box 1191, Texas City, Texas.

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